CTOBER 2, 1961
Volume 50, No. 20

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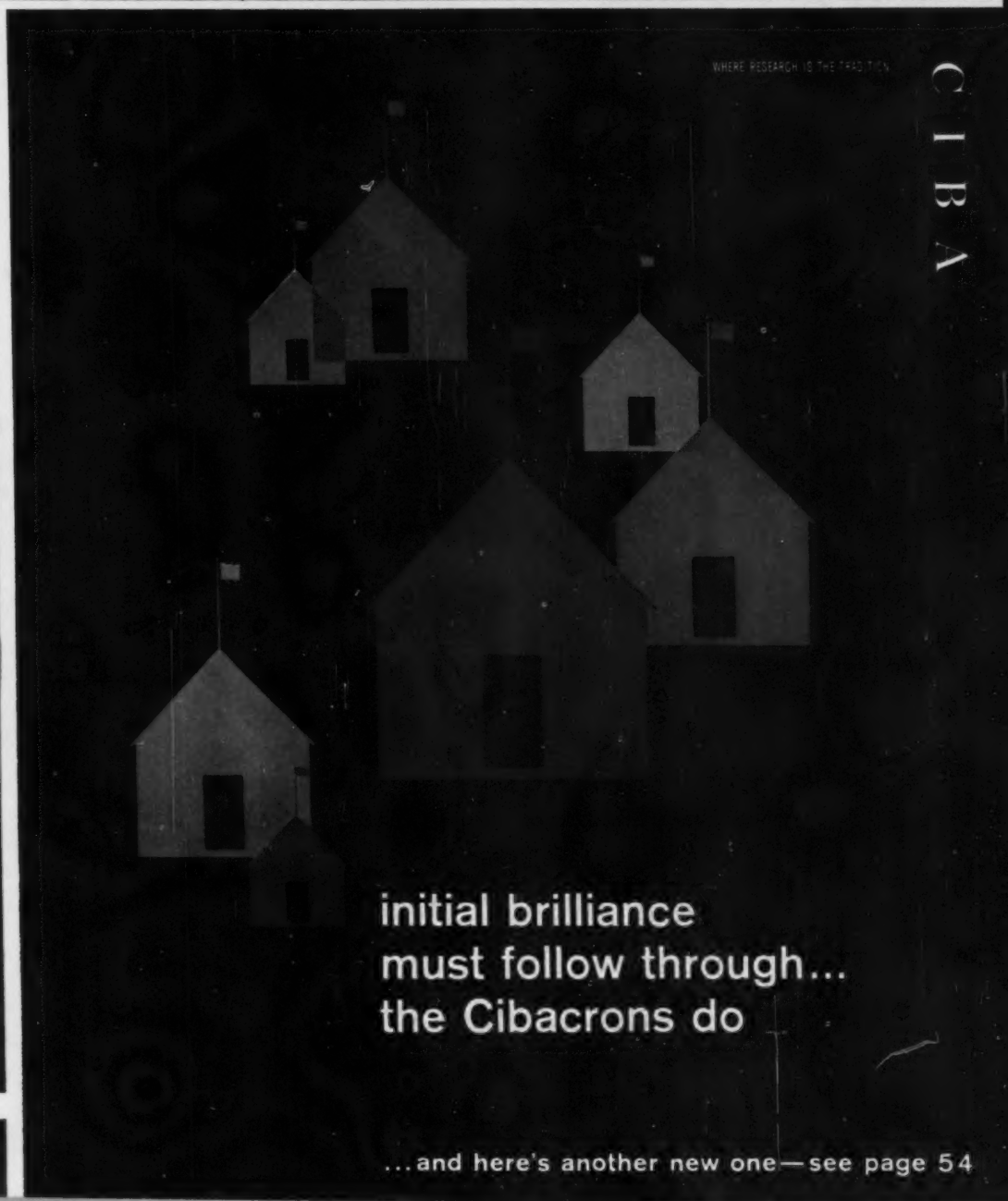
OFFICIAL PUBLICATION OF THE PROCEEDINGS AMERICAN ASSOCIATION OF TEXTILE CHEMISTS AND COLORISTS

AMERICAN DYESTUFF REPORTER

TEXTILE WET-PROCESSING



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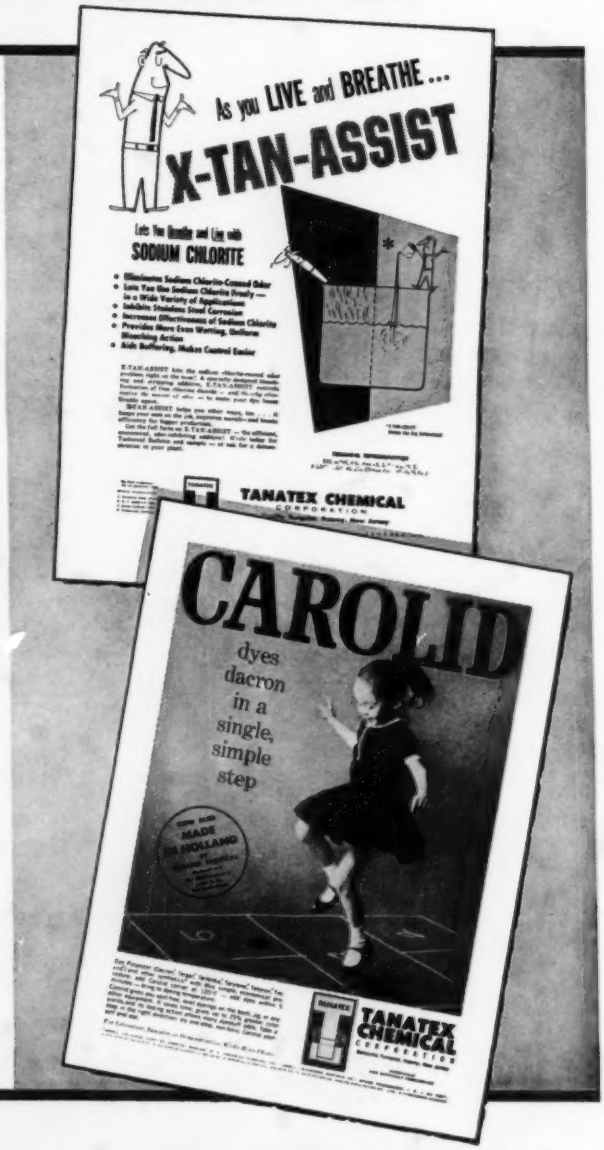

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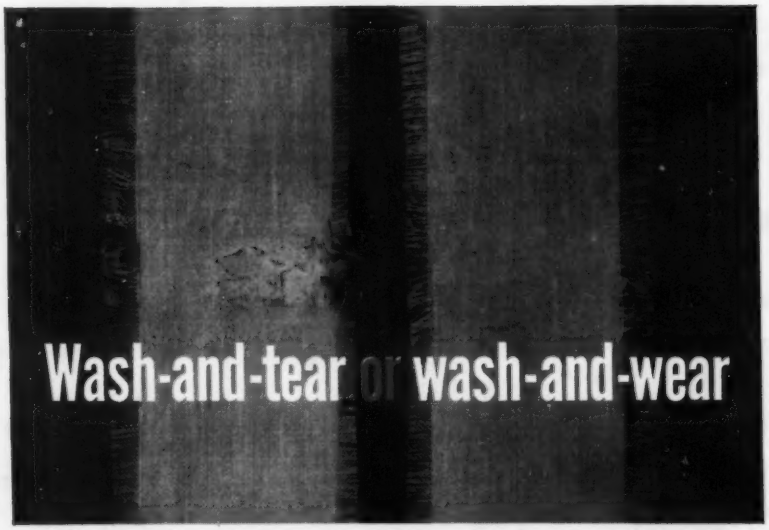

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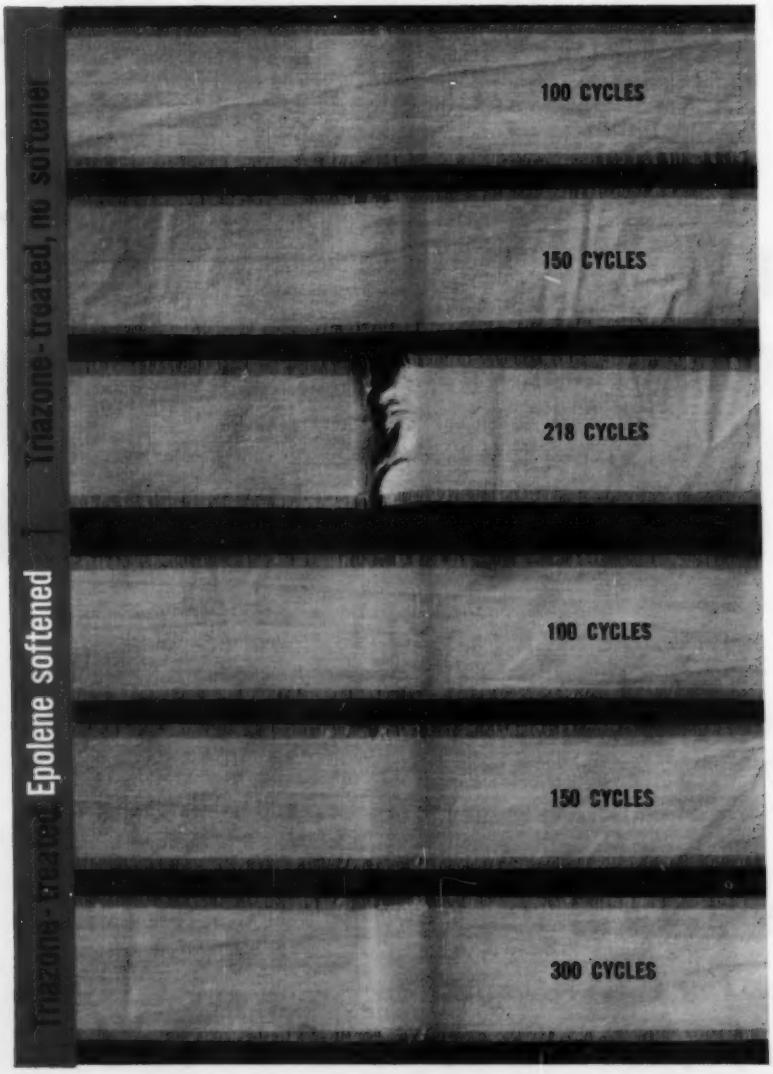

IT is axiomatic in the textile industry that the finishing resin alone does not a wash-and-wear fabric make. In fact, the variables are many, and often complex.

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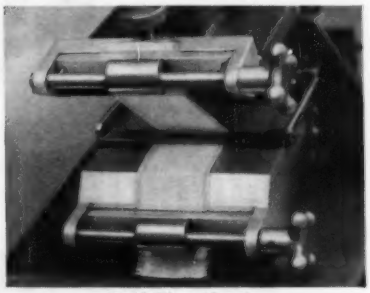

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RIZZO, RAMSLEY RECEIVE ELEVENTH AMERICAN DYESTUFF REPORTER AWARD

FRANK J RIZZO

ALVIN O RAMSLEY

OUR congratulations to Frank J Rizzo and Alvin O Ramsley of the Quartermaster Research and Engineering Command, US Army, Natick, Mass, on their selection for the 1961 AMERICAN DYESTUFF REPORTER AWARD.

The Award was presented to the two men at the recently concluded 1961 Convention of the American Association of Textile Chemists and Colorists at Buffalo, NY.

The paper selected for the 1961 Award, "New Color-measuring Instruments for Use by the Textile Industry", was delivered by Mr Rizzo at the 1960 AATCC Convention in Philadelphia on October 8, 1961. The paper appeared in the *Proceedings of the American Association of Textile Chemists and Colorists* in the May 15, 1961 issue of *American Dyestuff Reporter*, pages 53-63.

Judges for this year's contest, appointed by the AATCC Publications Committee, were: Paul B Stam, J P Stevens & Co, chairman; Charles R Williams, Monsanto Chemical Co; and Victor S Salvin, Celanese Fibers Co.

The Award was endowed by the Howes Publishing Company, Inc, as a memorial to Alfred P Howes, founder of the *American Dyestuff Reporter*. Its purpose is to encourage the presentation of the best possible papers before local and national meetings of the American Association of Textile Chemists and Colorists. The Award, $200 annually, is presented to the author or authors of the paper considered best in the opinion of the judges. Any technical paper presented before a local or national meeting of the AATCC and published in the *Proceedings* as they appear in the *American Dyestuff Reporter* is eligible with the following exceptions: Intersectional Technical Paper Competition papers, papers by employees of the AATCC, reports of research committees. It is a condition of eligibility that the paper have its first publication in the *Proceedings*. Papers published in the *Proceedings* from July 1st of one year to June 30th of the next year are considered in each judging period.

(continued on page 41)

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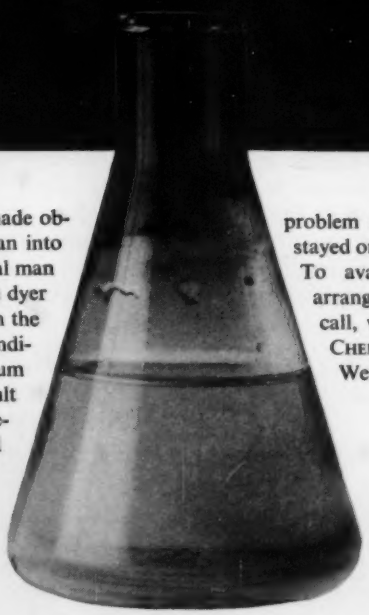

A dyehouse accepted an order calling for a shade obtainable only with a very sensitive dye and ran into trouble. We got an SOS. A "Virginia" technical man was sent around to see what he could do. The dyer was using test papers and titration to check on the potential of the bath. Under-reduction was indicated, so the dyer was increasing the sodium hydro and getting over-reduction. The end-result was either crocking or dull shades. Our trouble-shooter put his Redox recorder to work and found that the hard-to-handle dye stayed effectively soluble in approximately a 25 millivolt range. A new bath was prepared. By taking millivolt readings every 15 minutes, the exact proportions of hydro and caustic were finally established. To make sure the problem really had been solved, the "Virginia" man stayed on the job until the entire order was completed. To avail yourself of our technical service—or arrange for a free mill trial of "Virginia" hydro—call, wire or write: Chemical Division, VIRGINIA CHEMICALS & SMELTING COMPANY, Dept. 614, West Norfolk, Virginia.

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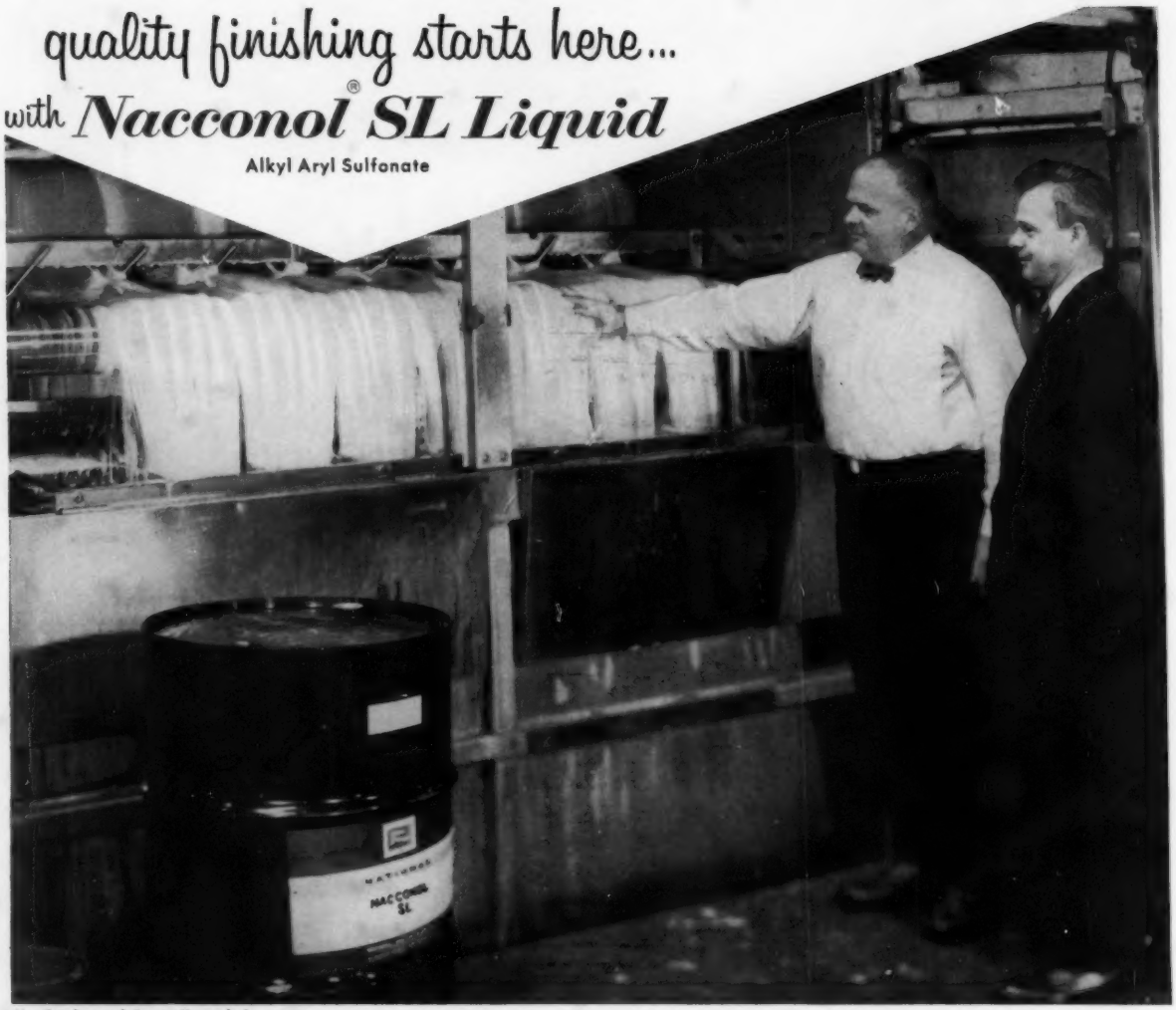

Mr. Stoehr explains to Kenneth Jones, National Aniline representative, why Botany prefers Nacconol SL.

write for this booklet!

says Richard G. Stoehr,
Vice President and General Manager
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PERMANENT ROTPROOFING OF CELLULOSIC FIBERS BY THE WET FIXATION OF RESIN PRECONDENSATES

ANDREAS RUPERTI

Research Laboratories, Ciba Ltd, Basle, Switzerland

This paper reports on the present status of cotton conservation by a wet fixation process using a water-insoluble condensation resin. This process, developed by Ciba, is said to offer insolubilization of resin within the fiber by wet fixation with no adverse effect on fiber properties. The principle of the process is discussed and the properties of cotton treated with a condensation resin are described. It attempts to show that this finish can be carried out with ease and safety on standard types of equipment. The possibility of providing cotton fibers with a highly durable protection against surface mildew growth by adding a suitable organic mercury compound to this finish is illustrated.

INTRODUCTION

THE problems involved in the permanent rotproofing of cellulosic fibers is sufficiently well known in a cotton-producing country, such as the USA, to make any further reference to its importance superfluous. The rapid spread of man-made fibers, which have inherent rotproof characteristics, has stimulated the interest of the cotton-finishing world in this problem, since it has become vital for the cotton fiber to be provided with a similar degree of rot resistance.

There are two conceivable methods of protecting cellulosic fibers from bacterial and fungal attack; these differ from each other in principle. Either the substance of the fiber can be modified so that it will not be used for food by bacteria and fungi, or an attempt can be made to destroy the microorganisms themselves. The conservation method, by which a defense barrier is formed without actively combating bacterial and fungal growth, is a passive form of protection. An active form of protection is the combating of microorganisms with substances that kill them or inhibit their growth; this is the way in which most conventional conservation methods work.

The use of active substances does not provide durable protection. Their durability is always impaired by a certain degree of solubility, a prerequisite for their activity which results in the leaching of these substances from the treated fabric; methods of passive protection, therefore, have been employed. Efforts have been made to render the fiber resistant to attack by converting it into a cellulose derivative by esterification or etherification. These derivatives are not liable to attack by microorganisms. Fabrics made from acetylated and cyanoethylated cotton have been offered commercially, but the chemical processing of cotton has shown no sign of becoming a major commercial development. Enclosed equipment manufactured from acid-resistant material is needed, since these processes have to be carried out in an organic medium, using volatile and toxic chemicals. Regeneration of chemicals is essential. The high cost of the process and suitable equipment means that one important advantage of cotton, ie, its cheapness, is lost completely.

Cotton can be rendered rotproof in a much easier way by depositing water-insoluble condensation resins in the fiber. This possibility (*1*) has been known for a long time, only no real use has been made of it. Conventional methods of applying condensation resins to cotton impair the mechanical properties of the fiber. Loss of strength is always undesirable. If large amounts of resin have to be applied, 15 to 25% owf being necessary for permanent rotproofing by conventional methods (*2*), intolerable losses in strength are incurred.

DISCUSSION

Ciba has succeeded in making fundamental progress in this area of finishing. It has developed a wet fixation process[1] using Arigal C condensation resin for the permanent conservation of cotton fabrics (*3*). In this process the mechanical properties of the fiber remain unimpaired and relatively lower amounts of the resin are used.

The principle is as follows:

A synthetic resin is deposited in the fiber; the resin is not cured in the ordinary way, but fixed wet in the presence of water. The insolubilization of the resin within the fiber provides a permanent barrier against microorganisms. No reaction takes place with cellulose and the properties of the fabric are hardly affected. The water absorption capacity of the fiber remains unchanged, retaining its affinity for direct dyes and having added affinity for acid dyes because of its resin content. Its dry crease resistance undergoes only a slight improvement, its tensile strength remains unchanged, and its abrasion resistance increases in most instances.

That cellulosic fibers treated with condensation resins should possess these properties must appear very surprising. It should, however, be borne in mind that the resin is formed when the fiber is in a swollen state. American research workers have shown how the properties of the finished fiber are a function of the state of swelling in their experiments with formaldehyde (*4*).

[1] US Patent 2,763,574; Swiss Patent 304,005

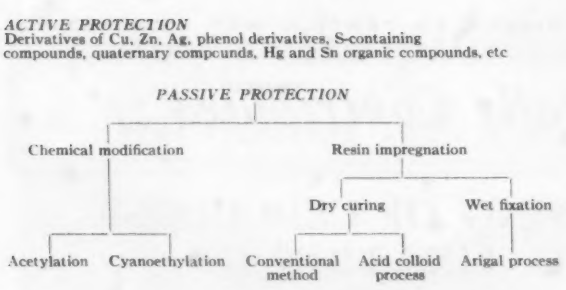

Figure 1
Conservation of cellulosic textiles

Furthermore, the water present in wet fixation hinders the crosslinking of the cellulose to such an extent that it hardly occurs in condensation-resin finishing. Some crosslinking can be obtained in wet fixation by working at higher temperatures or lengthening the reaction time. This would lead to the usual modifications in the properties of the treated fabric and cause loss of strength. Crosslinkage is not necessary for conservation, since insolubilized resin provides an adequate mechanical barrier against microorganisms.

The original form of treatment could be carried out only on special equipment provided with reaction chambers; this type of equipment is rarely found in textile plants. These difficulties appeared to be so serious that a team of American research workers proposed carrying out resin conservation by an acid colloid process, using conventional methods of curing and disregarding the detrimental effect on the mechanical properties of the fiber (5). The chief advantage of their acid colloid process was that it permitted continuous processing on conventional equipment.

Meanwhile, the technological side of wet fixation has been studied carefully with the result that cotton fabric can be treated quite simply and safely on standard types of equipment by taking into consideration certain peculiarities of this process.

The cotton fabric can be treated either semicontinuously by a wet aging process at room temperature or at a slightly higher temperature or continuously by treating it on a conventional continuous steamer.

The main obstacle to processing on standard equipment was the risk that the condensation-resin solution would migrate during wet fixation. Suitable measure had to be taken to overcome this difficulty: very high rates of expression and higher concentrations of condensation-resin solution have to be used, or the con-

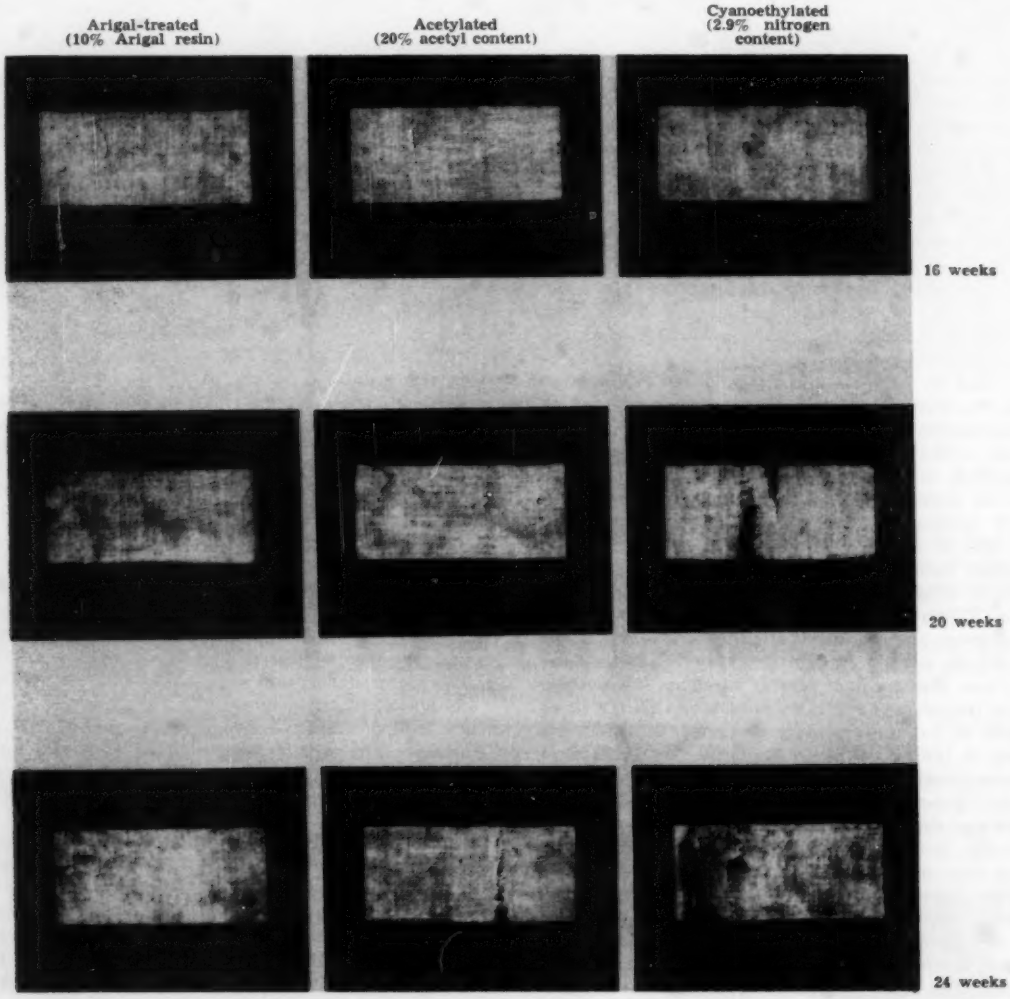

Figure 2

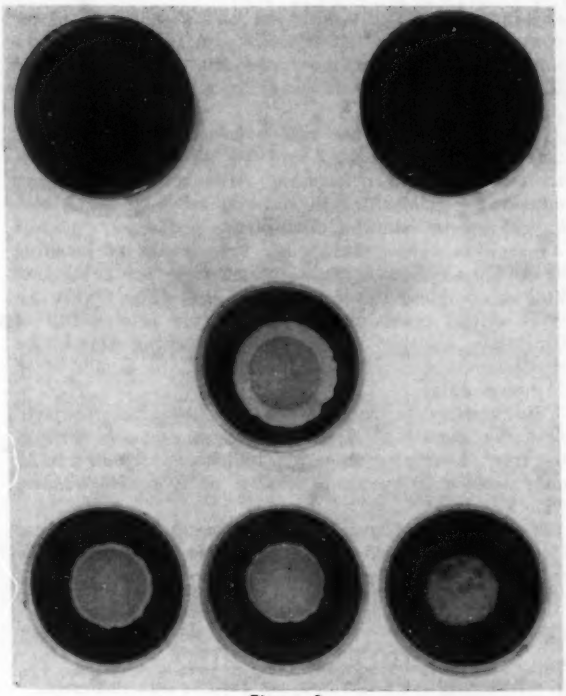

Figure 3
Top row: **(left) untreated; (right) 10% Arigal C**
Center: **9% Arigal C, 1% Fungitex 1165**
Bottom row: **(left) after seven days of leaching at 30°C; (middle) after 14 days of leaching at 30°C; (right) after 28 days of leaching at 30°C**

centration of the condensation-resin solution taken up by the fiber has to be increased by partial drying of the impregnated fabric. These measures prevent the resin from migrating and lead to a reduction of the curing time.

The risk that the resin will be caused to migrate in the wet fixation process is countered by early insolubilization of the condensation resin within the fiber. This can be achieved, for instance, by using small amounts of free acid, preferably formic acid, as catalysts. Since a reduction in the stability of the impregnation bath is caused by the use of free acid, a working compromise has to be found between the stability of the bath and the rate of insolubilization within the fiber. Another way is to apply the necessary acid to the impregnated fabric or to produce it on the fiber by chemical reaction.

These points have been duly considered in drawing up the following recommendations for the treatment of cotton fabrics by the wet fixation process with Arigal C condensation resin.

1) *Continuous Steam Process.* The fabric is padded with a solution of Arigal, to which five to 10 g/l hydrogen peroxide 30% have been added, and squeezed at a high rate of expression. If this is not possible the fabric should be dried on a tenter to a residual moisture content of 20 to 40%. The impregnated fabric is then steamed for at least eight minutes with saturated steam in a continuous ager. The heating-up of the impregnated fabric leads to the formation of formic acid by the oxidation of formaldehyde with hydrogen peroxide. The insolubilization of the resin within the fiber takes place very rapidly. The impregnation baths remain stable for several hours.

2) *Semicontinuous Wet Aging.* The fabric is impregnated with Arigal C and hydrogen peroxide, squeezed and, if necessary, partially dried to a residual moisture content of at least 20%. The fabric is then batched up in rolls, which are wrapped in plastic sheets to prevent them from drying. The rolls are kept either at room temperature or at a slightly higher temperature until the wet fixation process has been completed. It generally takes from one to four days, depending on temperature conditions.

3) *Formic Acid Process.* Hydrogen peroxide is replaced by small amounts of formic acid, which are not added to the impregnation bath but applied to the fabric after impregnation by spraying, nip-padding, etc. The resin is fixed wet by one of the methods previously described.

The amount of resin to be used depends on the type of fiber and the demands made on the treated fabrics. About 5% resin owf is sufficient to render cotton rotproof so that it will show no loss of strength after burial in compost at 30°C for six weeks. To be on the safe side, a somewhat higher amount of resin (approx 7% owf) is recommended. It is even advisable to use 11% Arigal C for goods having to meet the highest requirements, such as fishing nets or fabrics permanently exposed to weathering. Cotton conserved with such high amounts of resin is exceptionally resistant to strong bacterial and fungal attack. Figure 2 shows a comparison between fabrics treated with Arigal C and samples of chemically modified cotton (these samples were obtained commercially) in prolonged soil burial tests.

The Arigal treatment has a favorable effect on the resistance of cotton to weathering. A light cotton fabric permanently exposed without a glass cover to outdoor weathering for 12 months lost over 40% of its original strength through the action of light alone. The same fabric treated with Arigal C underwent only a 20% loss of strength on being weathered under identical conditions, and its rotproof characteristics remained unaffected.

A purely passive protection, irrespective of whether it was produced by treatment with Arigal C or chemical processing, prevents the development of microorganisms that feed on the actual substance of the fiber, but it cannot inhibit the surface growth of mildew whose food is drawn from other sources. Since the finisher is concerned not only with preserving the mechanical properties of a fiber but also with ensuring the aesthetic appearance of the fabric, growth of mildew cannot be tolerated, even if the fiber itself has not undergone any attack.

The use of an active substance in wet fixation with Arigal C is, therefore, often desirable. The durability of the mildew protection should not lag too far behind that of the rotproof conservation. The outcome of a long series of trials was that only certain organic mercury compounds were able to meet these requirements. If used in the wet fixation process with Arigal C, their leachability is greatly reduced but their effectiveness is preserved. It is, therefore, possible to obtain long-lasting mildew protection by using these compounds; they are harmless to users, since only rather low amounts are applied.

Ciba offers a solution of a suitable mercury compound to the trade under the provisional designation of Fungitex 1165. This product has been specially developed for use in the wet fixation process with Arigal C and is a viscous mass containing approx 3% mercury in

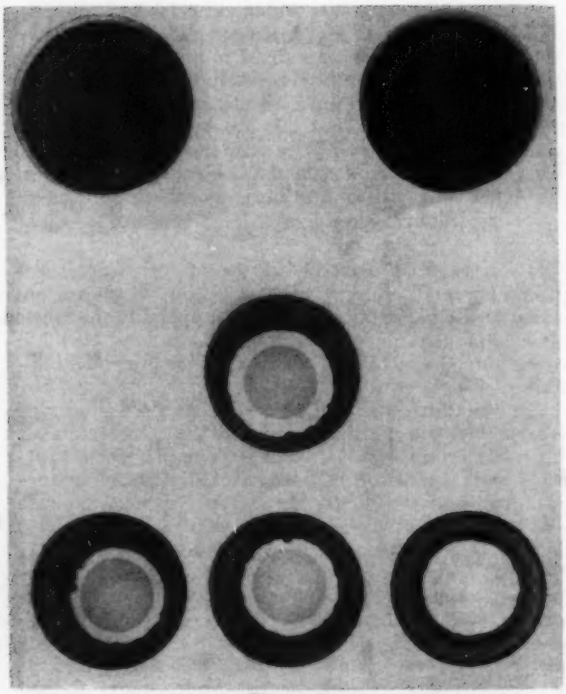

Figure 4

Top row: **(left) untreated; (right) 10% Arigal C**
Center: **9% Arigal C, 1% Fungitex 1165**
Bottom row: **(left) 9% Arigal C and 1% Fungitex 1165 after 30 washes—0.5% soap, 30 minutes at 40°C**
(middle) 9% Arigal C and 1% Fungitex 1165 after 20 washes—0.5% soap, 0.2% soda, 30 minutes at 60°C
(right) 9% Arigal C and 1% Fungitex 1165 after 10 washes—0.5% soap, 0.2% soda, 30 minutes at 95°C

organic form. It is readily soluble when added to the concentrated Arigal solution. It can be regarded as a modified Arigal component, since it is insolubilized in the wet fixation process and helps to produce a passive protection.

The durability of the mildew protection produced by using 1% Fungitex 1165 and 9% Arigal C is demonstrated by prolonged leaching. After seven and 14 days of continuous leaching in running water at 30°C, biological tests showed complete protection against *Aspergillus niger*. Only after four weeks of leaching at 30°C was their a slight reduction in the protection but no complete loss of fungitoxicity. (See Figure 3). (NB official procedure for testing the leachability of fungistatic finishes specifies that leaching should be carried out in running water at 30°C for a period of 24 hours only).

Similar results were obtained in tests for washfastness. No growth of *Aspergillus niger* was observed on the treated samples after they had been subjected to 30 mild washes, 20 moderate washes, or 10 severe washes. (See Figure 4).

It can be seen that a combined treatment with Arigal C and Fungitex 1165 offers a means for complying with consumer wishes for durably rotproofed and mildew-resistant cotton fabrics.

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TRADEMARKS

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ADR ABSTRACTS

The Stability of Polyvinyl Chloride: Its Laboratory Evaluation, and Evaluation Techniques for Stabilizers

Burdess, G D and Finley, C N, *Fibres & Plastics* 22, #1, 21-3; #3, 73-5, Jan and Mar 1961

The manufacture and use of materials based on polyvinyl chloride (PVC) resins have expanded considerably in recent years. One of the most important factors governing this expansion has been the parallel development of improved stabilizers; materials permitting the processing and end use of PVC under conditions which hitherto would have led to excessive degradation by heat and light.

The authors outline the reasons for the use of these stabilizers, the types of compounds employed, and the methods used for their laboratory evaluation.

When PVC is heated, decomposition begins at *ca* 70°C and hydrogen chloride is evolved. Following this, color changes ranging through yellow, brown to black occur, and finally embrittlement.

The degradation of PVC due to the action of light, especially radiation in the u v range, is not so quickly evident. It differs in a major way from that produced by heat. U v radiation affects properties such as tensile strength and plasticizer compatibility long before color formation becomes a problem, whereas under the action of heat the reverse is the case.

The action of both heat and light brings about cross-linking and chain scission which will affect the physical properties of the polymer. To counteract the effects of both heat and light, various compounds are added to the resins.

The ideal stabilizer should have the ability to react with degradation products (eg, hydrogen chloride) without increasing the rate of evolution, have antioxidant properties, and act as a screen against the penetration of u v radiation, to mention only a few of the requirements.

Of the many compounds proposed as stabilizers for PVC, only a few are commercially important. The metal soaps of the long-chain fatty acids are widely used, the acids employed being normally stearic and lauric. Of the many metal-containing substances suggested, compounds of calcium are the best-known of the nontoxic stabilizers. Among the various organic compounds proposed, those containing the "epoxy" group are the most widely used.

(continued on page 51)

PAPIN AND HIS NEW DIGESTER

"INVENTIONS OF THE PRESSURE KIER AND PRESSURE DYEING"

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IN John Evelyn's diary there is an interesting account of a supper that he attended with other Fellows of the Royal Society of London in 1684 (*1*).

> "Went this afternoone with severall of the R. S. to a supper, which was all dress'd, both fish and flesh, in M. Papin's Digesters, by which the hardest bones of beef itselfe, and mutton, were made as soft as cheese, without water or other liquor, and with lesse than eight ounces of coales, producing an incredible quantity of gravy; and, for close of all, a jelly made of the bones of beef, the best for clearness and good relish, and the most delicious that I have ever seen or tasted. We eat pike and other fish-bones, and all without impediment: but nothing exceeded the pigeons, which tasted just as if bak'd in a pie: all these being stewed in their own juice, without any addition of water, save what swam about the Digester, as in balneo; the natural juice of all these provisions acting on the grosser substances, reduc'd the hardest bones to tendernesse. This philosophical supper caus'd much mirth amongst us, and exceedingly pleas'd all the company. I sent a glasse of the jelley to my wife, to the reproach of all that the ladies ever made of the best hart-shorn."

On November 15th, 1800, John Turnbull Jr, was granted English patent #2449 (*2*) for—

> "new processes or improvements of processes and apparatus applicable to the bleaching or whitening, and to the purifying washing and cleansing of cotton, flax, hemp, silk and wool, and of goods of every description made or manufactured of cotton, etc."

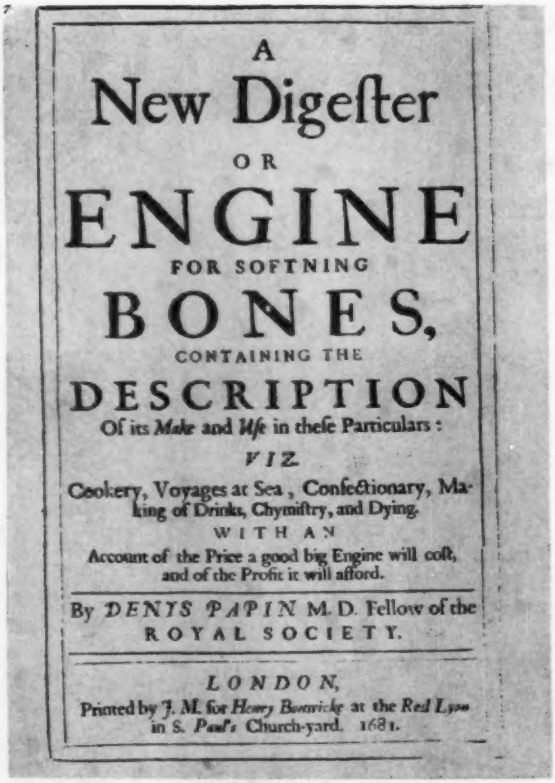
Turnbull's invention consisted of immersing "goods" in an alkaline solution and then steaming the "goods" in a closed vessel of sufficient strength and of proper shape and kept at a proper heat by means of a fire put under it so that—

> "By this process the goods can be heated considerably above the boiling point, a circumstance that adds so much to the dissolving power of the alkaline, or soaping, or other lye, that the quantity left in the goods after draining is found sufficient to dissolve and discharge by one steaming as much of a resinous, gummy, or other impurities from the goods under process as could have been discharged by long boiling of the goods in the lye itself."

Now, what is the connection between a dinner and a process for boiling out cotton under pressure? Denis Papin and his digester. Papin was the first to make practical use of the fact that, under pressure, the temperature of water could be raised above 212°F and thereby the solvent and chemical actions of water solutions could be improved. Papin's digesters were the prototypes of the kier, the sterilizer, the autoclave, and of many other pieces of apparatus which have meant so much to the textile industry, to the chemical industry and to our industrial civilization. Additional questions may now be posed. Who was Papin; what is the story of his invention, and how does Turnbull's patent fit into the matter?

Illustration 1
Engraved portrait of Papin with his "machines"

Denis Papin, MD, Fellow of the Royal Society, was born August 22, 1647, in the town of Blois in France. Papin's father was a medical doctor and his family was Protestant. Papin probably obtained his degree in medicine at Orleans. Shortly thereafter he came to Paris and was fortunate to find employment with the famous Dutch physicist, Huygens. His experiments with Huygens were concerned with the use of expanding "air" from burning gunpowder for doing useful work. Apparently, Papin's future field of work was delineated at this time. For the remainder of his life his efforts were concerned mainly with the relationships of the pressure and temperature of air and water and the application of these relationships to useful purposes. Papin's first paper in this field was published in Paris in 1674 under the title "New Experiences con-



A
New Digeſter
OR
ENGINE
FOR SOFTNING
BONES,
CONTAINING THE
DESCRIPTION
Of its *Make* and *Uſe* in theſe Particulars:
VIZ.
Cookery, Voyages at Sea, Confectionary, Making of Drinks, Chymiſtry, and Dying.
WITH AN
Account of the Price a good big Engine will coſt, and of the Profit it will afford.
By *DENYS PAPIN* M. D. Fellow of the ROYAL SOCIETY.
LONDON,
Printed by J. M. for *Henry Bonwicke* at the *Red Lyon* in S. *Paul's* Church-yard. 1681.

Illustration 2
Title page of first edition of Papin's book on "The Digester"

cerning the vacuum with a description of the machine for making it." (3).

In 1675 he went to England and worked for several years with the famous Robert Boyle on the latter's "pneumatic machine." By 1680 the young Frenchman had been made a member of the Royal Society. It was at this period that Papin began his studies on the effects of steam under pressure and the experiments with his digester were often exhibited during the meetings of the Royal Society. These experiments with the digester were of such great interest to the Royal Society that it requested that they be written up and published. (4). They appeared in London in 1681 under the title, "A New Digester or Engine for softening Bones, containing the description of its make and use in these particulars, viz. Cookery, Voyages at Sea, Confectionary, Making of Drinks, Chymistry, and Dying. With an Account of the Price a good big Engine will cost, and of the Profit it will afford." (See Illus 2).

We need not be surprised to see Papin giving prominence in the title to the use of his "bone digester" in dyeing. Papin was a practical man and, as we shall see, "pressure dyeing" was one of his inventions. Papin was not ashamed to be working in a field which was rather prosaic when compared with astronomy, mathematics and physics being studied by many of the Fellows of the Royal Society. The preface to his book is interesting from this aspect (5):

> "Having therefore reason to believe that this piece may fall into the hands of divers persons that would never read the history of the Royal Society, nor Mr. Boyle's book about the usefulness of experimental philosophy, I thought this to be a very fit place to undeceive those that imagine it to be a folly to look for new discoveries, and that all things are already found out.

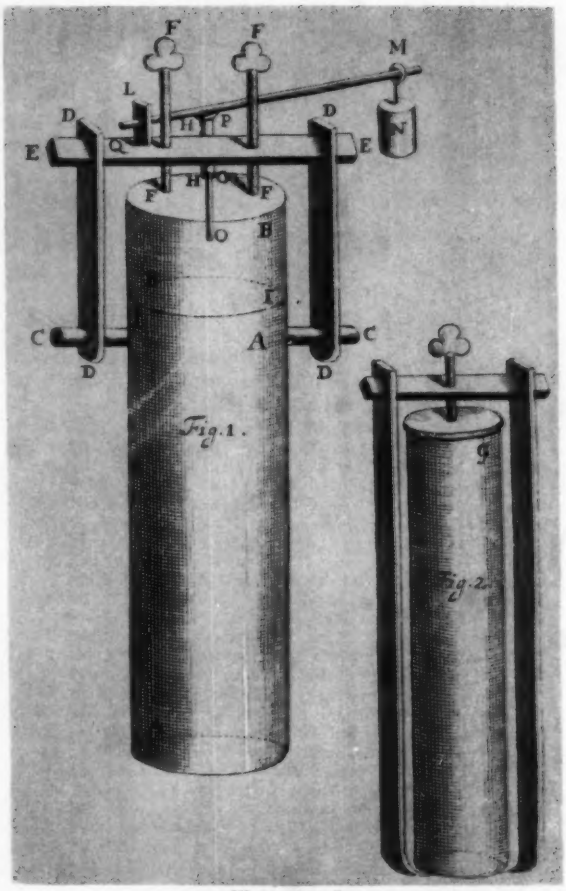


Illustration 3
Papin's digester showing safety valve and inner cylinder

> For the confuting this errour I will take nothing but from my subject, and even amongst several instances it affords I will use but one. Therefore I shall only say that cookery is such an ancient art, the use whereof is so general and so frequent, and people have been so earnest upon the improving of it, that it seems if any could be brought to perfection, this should be it: nevertheless nobody can deny but it will be now considerably improved, seeing by the help of the engine here treated of, the oldest and hardest cow beef may be made as tender and as savory as young and choice meat. I may besides say, that this was no hard matter; for everyone knows that compact bodies, if hot, will burn more powerfully than those of a more rare contexture: that red hot iron, for example, will do more than coals: so there was no question but that water being heated enough for boiling, and shut up so as not to be able to expand itself, as by ebullition it doth, such water, I say, would effect much more than if permitted to its ordinary boiling expansion. For my part, as soon as this came into my mind by making some experiments about compression for Mr. Boyle, I thought it so certain, that I made no question to undertake the tryal: yet though the thing was so easie, nobody that I know of, had any thoughts of it: many learned men have done and do still things of much more difficulty but nobody can see all things: therefore we must confess that there may still remain discoveries to be made by small as well as great capacities. And no man that industrially prosecutes a subject though seemingly never so trivial, need despair of obtaining that great felicity of

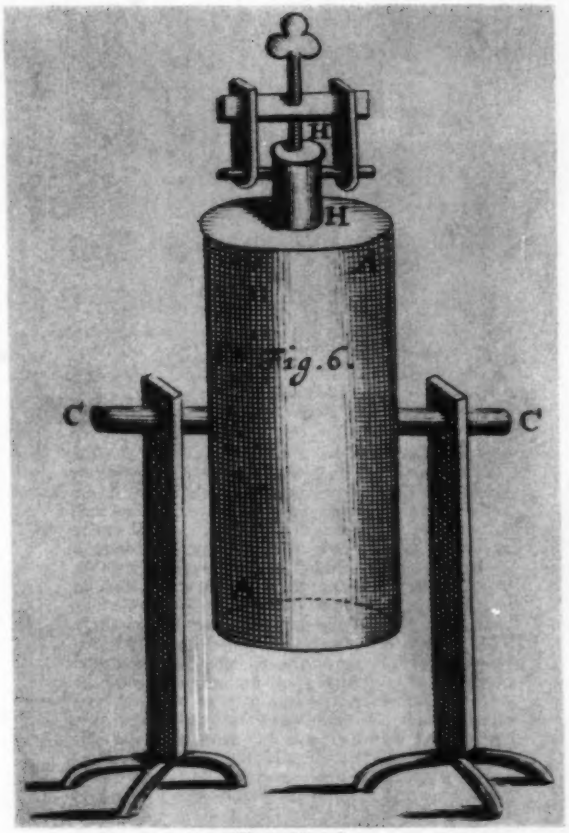


Illustration 4
Papin's pressure kier

having made some discovery, the usefulness whereof may recommend it to posterity."

Perhaps Papin's words can be an inspiration to those of us who are working in the prosaic textile industry and who sometimes feel that there are not the same possibilities of romantic discoveries as exist in the new fields of atomic energy and electronics.

Papin's pressure cooker was simple in principle but sophisticated in design. Illus 3 shows the component parts of Papin's digester as well as the safety valve which Papin also invented. The unit at the left in Illus 3 (ie, Fig. 1) shows the digester ready for heating. AA is a hollow brass cylinder shut at the bottom and open at the top. BB is a shorter hollow brass cylinder of the same diameter which fits together with the cylinder AA. The two cylinders are held tightly together by screwing down the screws FF. At II where the two cylinders come together there is a circular washer of paper which is kept moist and serves to make the joint tight. HH is a little pipe soldered to a hole in the cover DD and is fitted with a closely ground valve P. Paper is kept between P and HH to make the valve fit very tight. By moving the weight MN to different positions on the bar, the pressure within the kettle can be regulated.

The unit at the right in Illus 3 (ie, Fig. 2) is a smaller cylinder made of glass or metal and fitted with a cover held down with a screw. Items to be treated under pressure are closed up within this smaller cylinder, which is then placed within the apparatus of the unit at the left (ie, Fig. 1). Before the two cylinders AA and BB are locked together, enough water is placed in the lower cylinder AA to surround the smaller cylinder. After final sealing, the weight on the safety valve is adjusted and the machine is then heated.

While Papin could measure the pressure within the vessel by the setting of his saftey valve, he did not have a means of measuring the temperature within his vessel. He did try to obtain some measure of "the degree of heat" applied to his digester by the following method (6):

> "I hang a weight to a thread about three foot long, so that every swing makes about a second, and I let fall a drop of water into a little cavity made for that purpose at the top of it, and I tell how many times the hanging weight will move to and fro before the drop of water is quite evaporated: and I take care that the place where I put the drop may be clean because a little grease will considerably hinder its evaporating."

Now let us listen to the results of a few of Papin's experiments with his digester in his own words. In his chapter on "Experiments for Cooks" (7), he reports that, on July 13:

> "I took an old male and tame rabbet, which is ordinarily but a pitiful sort of meat: I seasoned it and put it into two glass pots: I kindled six ounces of coals, and prest the fire till the drop of water would evaporate in less than four seconds: the inward pressure was about six times stronger then the ordinary pressure of the air. The fire being gone out of itself I found the rabbet very ready and the bones softened, and it was as savoury as young rabbets use to be: the juyce of it turned to a good jelly: so that I think this to be the quantity of fire most fit to boil rabbets."

The problem of adequate food for ships at sea appealed to Papin and in Chapter III, "Experiments for Voyages at Sea," he says (8):

> "I took beef bones that had never been boiled but kept dry a long time, and of the hardest part of the leg: these being put into a little glass pot with water, I included in the engine, together with another little glass pot full with bones and water too, but in this the bones were ribs, and had been boiled already. Having prest the fire till the drop of water would dry away in three seconds and ten pressures, I took off the fire, and the vessels being cooled, I found very good jelly in both my pots; but that which was made out of ribs, had a kind of reddish colour, which, I believe, might proceed from the medullar part: the other jelly was without taste or colour, like harts-horn jelly; therefore I do not see any reason why it should not have the same effect; and I may say, that having seasoned it with sugar and juyce of lemmon, I did eat it with as much pleasure, and found it as stomachical as if it had been jelly of harts-horn."

Finally in Chapter VII entitled, "Experiments for Dyers," Papin reports on his inventions of pressure dyeing and of the textile kier (9). (See Illus 4).

> "I put two pieces of cloth into two glass pots; to one of which I added some tincture of coarse cochenille, and to the other juyce of prunes distilled after the manner described Chapter VI, Experiment 3. I prest the fire till the drop of water would dry away in 42 seconds, with an inward pressure six times stronger then the ordinary pressure of the air: then I took off the fire quickly for fear the cloth should be spoiled: the vessel being cool I found both the pieces of cloth good still and well dy'd, the juyce of prunes having penetrated as well as the tincture of cochinelle; but the tincture of it was of a deeper red and nearer to a brown colour: the juyce itself was much altered for it was violet before: it was grown also much more liquid and watry.
>
> "From this experiment it appears, that this engine keeping things for a great while in a great heat without damage, and hindring the most subtile parts from getting away, as usually

they do, may be fit to insinuate into cloth such liquors as are reckoned to be too thick and glutinous, as the juyce of plums is; because for dying there is no need of good taste.

"Mr. Mayre thinks there would be no need of an inward pot, and so I believe the aperture of the outward engine might be lesser than the cavity as you may see fig. 6, yet if they would dye cloth in it, the aperture HH should be left wide enough to convey the cloth into the cavity AA, and this engine should also hang in aequilibrio by its appendices CC for the convenience of filling and emptying the same."

Papin's design of the first kier embodies the same fundamental principles that are still employed today. While Papin used his kier for pressure dyeing, he clearly recognized that the increased action of solutions at high temperatures could be employed for other textile treatments.

Papin's fame became widespread particularly because of his work with "The Digester." In 1687 he was offered the chair of mathematics at Marbourg, and he left England to accept this position which he filled for some years. After leaving England, Papin was responsible for two other great inventions—the steam engine and the steam boat—inventions which are usually attributed to others. (*10*).

In 1690 Papin published a memoir entitled, "Description and Use of a new Machine to Raise Water." In this memoir he explained for the first time the theory of the steam engine which worked by the alternate action of a piston. Nothing came of this since the engine was not built.

Savary and Newcomen had erected their first steam engine in 1705 and this was based on the principles explained by Papin in his paper of 1690. Papin finally did build a steam engine based on his original principles and on some of the mechanical techniques of Savary and Newcomen for the Landgreve of Hesse. This was put to use raising water from the river Fulda (*11*).

Papin had also occupied himself for several years in constructing a boat operated by steam. This he achieved in 1704. Papin desired to show his boat to the Royal Society with the hopes that the Society would assist in raising funds to further his project. Papin did set sail in his steamboat on the River Fulda with the idea of steaming to a seaport where the whole boat could be transported to England. Papin's voyage was beset with many difficulties. When he reached the Weser River the boatmen who were jealous of their perogatives and fearful of eventually losing their jobs to this new steam boat, refused to let him continue on. He, his family and his possessions were removed from the boat and the boat was completely destroyed by the boatmen. Papin managed to get to London in dire circumstances and without his steamboat. (*3*). On the 11th of February, 1708, he submitted his proposition to the Royal Society "Concerning a new-invented boat to be rowed by oars, moved with heat." This proposal is recorded in the records of the Royal Society as follows (*12*):—

"It is certain that it is a thing of great consequence to be able to apply the force of fire for to save the labour of men; so that the Parliament of England granted, some years ago, a patent to Esquire Savery, for an Engine he had invented for that purpose; and his Highness Charles, Landgrave of Hesse, hath also caused several costly experiments to be made for the same designe. But the thing may be done several ways, and the Machine tryed at Cassell differs from the other in several particulars, which may afford a great difference in the quantity of the effect. It will be good, therefore, to find out clearly what can be done best in that matter, that those which will work about it may surely know the best way they are to choose. I am fully persuaded, that Esquire Savery is so well minded for the publick good, that he will desire as much as any body that this may be done.

"I do therefore offer, with all dutifull respect, to make here an Engine, after the same manner that has been practised at Cassell, and to fit it so that it may be applyed for the moving of ships. This Engine may be tryed for an hour or more, together with some one made after the Saveryan method. The quantity of the effect should be computed both by the quantity of water driven out of each Machine, and by the height the said water could ascend to. And to know the said height, we should use the method advised by the Illustrious President; viz. to try to throw bullets by the said Engine, with the inclination of forty-five degrees, and reckon that the said height is half the horizontal distance to which the bullets will be driven; and this would be the rule so well for one as for the other Machine.

"I wish I were in a condition to make the said Cassellian Engine at my own charges; but the state of my affairs doth not permit me to undertake it, unless the Royal Society be pleased to bear the expense of the Vessel called 'Retort', in the description printed at Cassell; but after that I will lay out what is necessary for the rest, and I will be content to lose that expense, in case the contrivance of the Landgrave of Cassell doth not as much again as that of Esquire Savary; but in case the effect be such as I do promise it, I do humbly beg that my expense, time, and pains, may be paid, and I reckon this to amount to 15 pounds sterling. If the Royal Society be pleased to honour me with their commands upon such conditions, the first thing to be done is to let me see the place where the Machine must be set, and I will work for it with all possible diligence; and I hope the effect will yet be much greater than I have sayd."

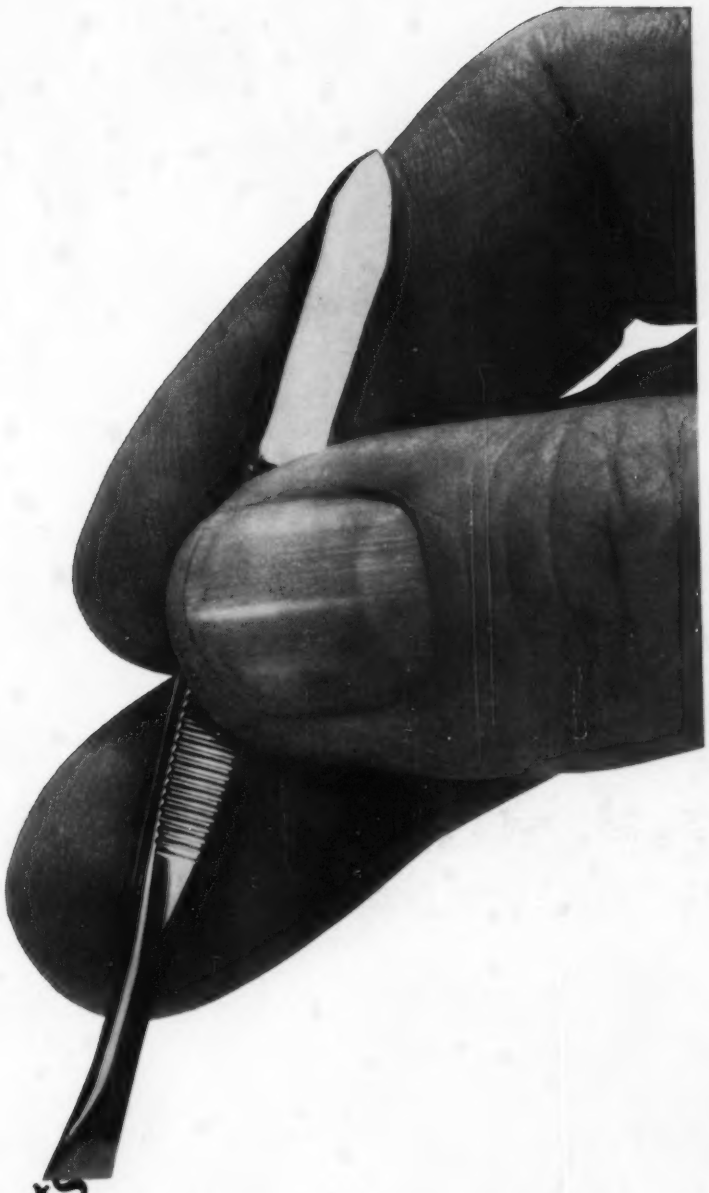
Illustration 5
Artist's conception of destruction of Papin's Steamboat

The records of the society did not show that any help was given to Papin. A second steamboat was never built. Papin's last days were probably spent in abject poverty on the continent. Records do indicate that he most likely returned to Cassell and died there in 1714. (*3*).

We now return to the discussion of the problem concerning the fact that while Papin in 1681 had given a clear description of a kier for treating textiles under pressure, this was not put into use until about 1800 and then only for alkaline boiling. Pressure dyeing itself has only come into its own within this century.

There are no records that we know of which can give the exact explanation as to why this was so. We can only attempt good guesses which are based on the available facts.

(*concluded on page 51*)



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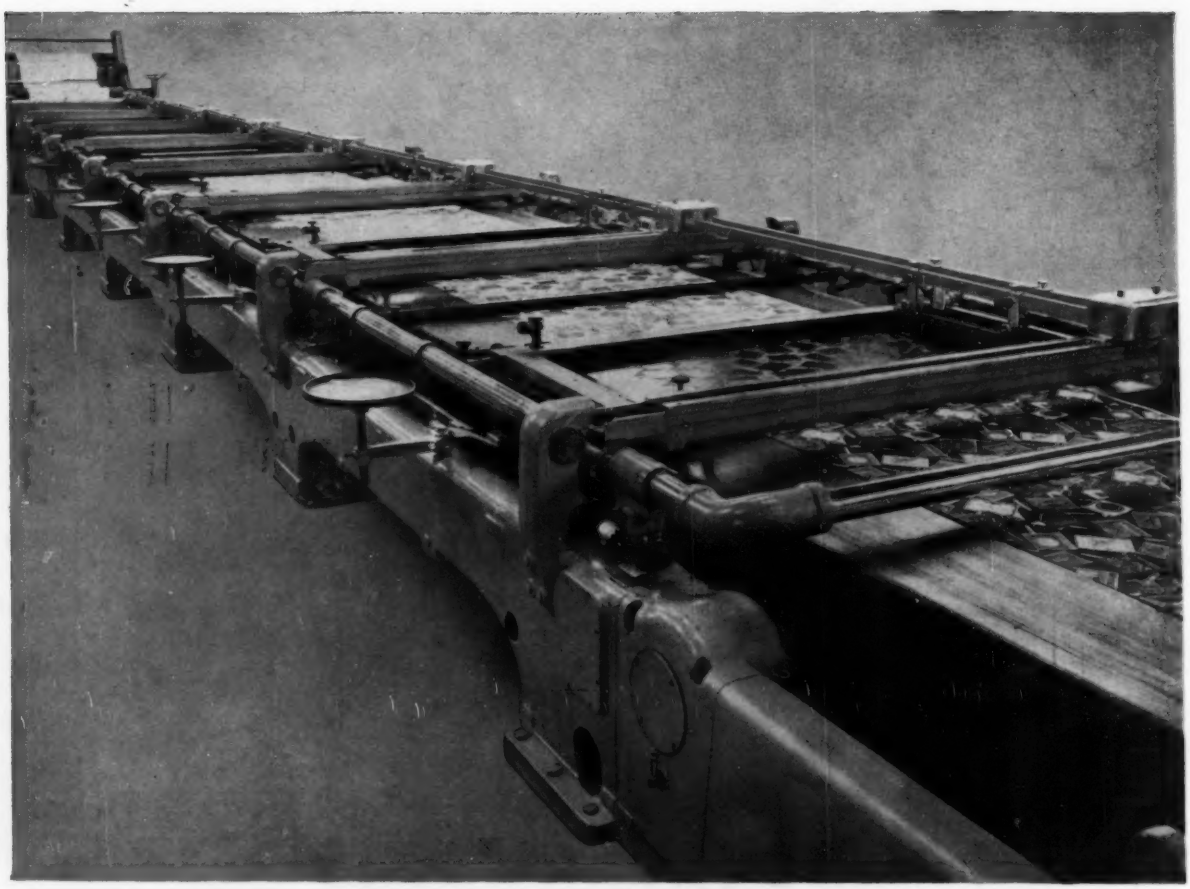

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1961 Convention———

FLUORESCENT WHITENING AGENTS IN "WASH-WEAR" FINISHING OF COTTON*

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The use of fluorescent whitening agents in the "wash-wear" finishing of cotton fabrics has been investigated. Three processing techniques for applying the whitener were used. Applications prior to the crosslinking treatment, after the crosslinking treatment, and with the crosslinking agent in the same pad bath were studied. Eight fluorescent whitening agents and two crosslinking agents were included in the work.

Color measurements indicating the whitening effects produced in the fabric by the different finishing treatments were made with ultraviolet radiation either included or excluded. Durability of these whitening effects to various types of laundering was determined. Any changes in the fabric's resistance to wrinkling and susceptibility to chlorine and light damage due to the treatments also were noted.

INTRODUCTION

FLUORESCENT whitening agents, often called optical bleaches or brighteners, have been used in the textile industry only since 1946, although they were available on the market for many years previously (*1*). The market today is over $15,000,000 per year and much research has been carried on in the use of these agents in soaps and synthetic detergents, paper, and on textile fibers of all kinds. However, much of this information is confidential or reserved for customer service and is not generally available.

Use of whiteners has become so general that it is difficult to buy a household laundering detergent which does not have one or more in it. They are used also in finished yard goods to such an extent that complaints have been made as to their indiscriminate use because of the different shades of "white" obtained (*2*) and changes in color (discolorations) during drycleaning (*3*). The entrance of "wash-wear" cottons and their tremendous growth in recent years has brought other problems into the picture. Most of the fluorescent agents used on cotton are essentially colorless dyes which "dye" the cotton in a manner similar to direct dyes. It is well known that the usual, crosslinked "wash-wear" cottons are so dye-resistant that whiteners are not efficiently taken up during laundering. In the treating of cotton fabrics with the usual commercial-type N-methylol crosslinking agents to produce a "wash-wear" finish, a degree of yellowness is often imparted to the fabric. This off-white appearance is considered very undesirable by the consumer who has been educated to associate a pure white finish with cleanliness. The finisher today is vitally concerned with methods of rectifying any off-white resulting from fabric processing.

The action of fluorescent whiteners was studied as a part of the Agricultural Research Service's broad research program on cotton. This paper reports the results of an investigation which was initiated in cooperation between research groups at the Southern Regional Research Laboratory and the Clothing and Housing Research Division. The information gained is of interest both to the cotton finisher and to the homemaker.

In preliminary trials with a typical whitener it was observed that application to cloth, followed by the "wash-wear" finishing agent, or simultaneous application with the agent and curing, both gave fabrics which were whiter than when the same brightener was applied to the finished cloth. This discovery seemed to be well worth developing. However, soon after the project was started, it was evident that the idea had occurred independently to a number of investigators. In a private communication, research on the subject was suggested by the research director of a large textile company; also, examination of "wash-wear" cottons from several manufacturers showed the presence of whiteners. Recently, a Japanese patent on the subject (*4*) shows that the idea was world wide. Nevertheless, finishers have shown great interest in the subject and the project was continued.

BACKGROUND

Fluorescent whitening agents improve the appearance of textile materials through two separate visual effects: a bluing effect and a lightening effect (*5*). These effects are due to absorption of ultraviolet radiation, which is invisible to the human eye, and reemission of this radiation as visible blue light. Thus, the whiteners compensate for the yellow color of off-white materials (*6*). This produces a white color without loss of total reflected light. In spite of the somewhat nebulous character of whiteness, a number of formulas have been developed for its measurement by combining the effects of grayness and yellowness. Studies of visual ratings have indicated that, for the same reduction in reflectance, yellowing reduces visual whiteness about four times as much as graying does (*7*).

The whitening agent can lose its fluorescent whitening ability on exposure to sunlight. If the exposure is not uniform a contrast between bright and dull causes the fabric to look dirty and yellowed (*2*). Recently reported work, however, indicates that natural sunlight exposure is not nearly as harmful to fabric finished with fluorescent whitening agents as artificial sunlight tests (*8*).

Villaume has shown that reduced whitening and fastness properties are

*Presented at the 1961 National Convention at Buffalo, NY, September 29, 1961.
[1]One of the laboratories of the Southern Utilization Research and Development Division, Agricultural Research Service, U S Department of Agriculture.
[2]Agricultural Research Service, U S Department of Agriculture.

obtained when fluorescent whitening agents are used on resin-treated cottons as compared with untreated cotton (*9*). Furry and coworkers have shown that fluorescent whiteners included in commercially available household laundry detergents produced less whitening effect on resin-treated cotton and the effectiveness varied with the finish (*10*).

A number of articles have been published which cover the nature of fluorescent whitening agents, historical development, chemistry, properties, and uses (*11-14*). A list of fluorescent whitening agents indicating the trade name, manufacturer, chemical composition, fiber substantivity, fluorescence properties, lightfastness limitations, and methods of application has been published (15). There are, however, hundreds or even thousands of agents which have not been tabulated in this manner.

MATERIALS AND METHODS

The fabric used in this study was an 80 × 80 white cotton print cloth (3.2 oz/sq yd), which had been desized, caustic-boiled, and bleached. Wrinkle-resistance agents used were dimethylol ethyleneurea (DMEU) and dimethylol ethyltriazone (DMET), which were obtained from commercial sources as 50% aqueous solutions. Zinc nitrate was used as catalyst in the application of these wrinkle-resistance agents.

Eight fluorescent whitening agents, which represented the principal classes of such agents, were selected for study from a range of available commercial products. The fluorescent agents included in this paper are listed in Table I with their code designation, their class, and their substantivity to textile fibers.

Fluorescent whitening agent and wrinkle-resistance agent were applied to the fabric in both laboratory-scale batch treatments and semipilot-plant-scale continuous treatments. The scheme of processing steps employed in both methods and the designation of the procedure used in the paper are:

I. Fabric treated with fluorescent whitener only.
II. Fabric treated first with fluorescent whitener and then with wrinkle-resistance agent in a a second step.
III. Fabric treated first with wrinkle-resistance agent and then with fluorescent whitener in a second step.
IV. Fabric treated with wrinkle-resistance agent and fluorescent whitener in a one-step operation. The two agents and catalyst were applied from the same pad bath before the curing step.

TABLE I
Fluorescent whitening agents used in this study

Designation Used[1]	*General chemical class of whitener*	*Fibers substantive to whitener*
A	Stilbene derivative	Cellulosic
B	Stilbene derivative	Cellulosic
C	Triazinyl diaminostilbene derivative	Cellulosic and nylon
D	Aminocoumarin derivative	Wool, silk, nylon, and acetate
E	Benzidinesulfone derivative	Primarily cellulosic but also acrylic and nylon
F	Pyrazolone derivative	Wool and nylon
G	Benzimidazole derivative	Cellulosic and nylon
H	Benzoxazole derivative	Acetate, triacetate, cellulosic, nylon, polyester and polyvinyl chloride

[1] The whiteners used were commercial products; however, they are not further identified by agreement with manufacturers who generously supplied samples and provided confidential information as to chemical classification.

Concentrations of agents and catalyst used (weight percent of the treatment bath) were: DMEU or DMET, 7%; zinc nitrate hexahydrate catalyst, 0.5%; and fluorescent whitening agent, 0.1%.

In the laboratory, swatches of fabric, approximately 12 inches square, were treated in duplicate. The fabric was immersed in the treating bath, and the wet pickup adjusted to approximately 80% by passing through pad rolls. It was then mounted on pin frames at original dimensions and dried at 140°F for seven minutes. For treatments which included wrinkle-resistance agents, the fabric was cured at 320°F for three minutes following the drying step. After treatment, samples were washed with a slightly alkaline solution of nonionic detergent and dried. Samples treated with different whitening agents were not mixed in the washing operation.

In the pilot plant, samples of fabric, approximately 15 inches wide and 20 yards in length, were finished on a narrow-width continuous finishing range with padder, drying oven, and curing oven in tandem. Drying conditions were 170°F for seven minutes; curing conditions, 330°F for two minutes and 20 seconds. For samples in which the curing step was not needed, passage through the curing oven was eliminated. After being finished, samples were washing on a jig with an alkaline solution of nonionic detergent, and dried.

Launderings were carried out in a household-type automatic washer using the regular cotton wash cycle. Wash solution was varied as indicated in the text. Samples were tumble-dried after each cycle for 20 minutes at 190°F. Some launderings also were made along with white shirts at a New Orleans commercial laundry which used the washing procedure suggested by the American Institute of Laundering (*16*). Samples were also washed in accordance with an AATCC severe wash procedure (*17*). The effect of stringent acid scouring conditions were tested by laundering a set of samples three times using the "delicate" fabric cycle of the automatic washer and ending each wash cycle with a rinsing at pH 3.4. Another set was treated with a solution containing 0.5% soap and 0.2% soda ash at the boil for one hour.

Determinations of physical properties of fabrics were performed by standard test methods of the ASTM (*18*). Damage due to retained chlorine was determined by the AATCC "scorch test" method (*19*). The results of this test are reported as the percentage of the original strength retained after chlorinating and scorching.

The Fade-Ometer[2] and the Weather-Ometer were also used in the evaluation of samples.

Quantitative measurements of the whitening effects were carried out using two instruments. The Hunter Color and Color Difference Meter (*20*) was adapted for use with or without a Noviol Shade A filter to obtain fluorescent and nonfluorescent whitening effects. This instrument gives three values for each measurement: R_d, or the percentage of light reflectance from 0 to 100; a, which denotes redness when plus, gray at 0, and greenness when minus; and b, which denotes yellowness when plus, gray at 0, and blueness when minus. A perfect white would have 100% reflectance with no chromaticity, that is, both a and b would be zero.

Blue reflectance was measured on a fluorescence-sensitive reflectometer (*21*). Reflectances can be made

[2] Throughout this paper, the mention of trade names and firms does not imply their endorsement by the Department of Agriculture over similar products or firms not mentioned.

on this instrument with ultraviolet radiation either included or excluded and offer a simplified measure of whiteness by combining in one figure the grayness and yellowness departures from ideal whiteness.

EXPERIMENTAL AND RESULTS

USE OF VARIOUS TYPES OF WHITENERS——Samples of cloth were treated with DMEU and eight different fluorescent whiteners applied by three different finishing techniques as detailed in the previous section. The results are shown in Table II. Included, of course, for comparison are the untreated fabric and fabrics treated with DMEU and with the eight whiteners alone. The characteristics of the latter set may be different from those produced by the more usual dye exhaustion process. These should be considered as controls for the applications with DMEU. Pad-bake processes have been proposed, however, for the application of certain whitening agents to synthetic fibers (*22*).

Color effects of the untreated cotton were practically the same with ultraviolet radiation included or excluded, indicating no fluorescence. Blue reflectance values of the sample also were practically the same under these lighting conditions. Both the color measurements and the blue reflectance values indicated slight increases in yellowness due to the DMEU treatment.

In general, all the other samples, which were treated in some way with whiteners, showed some fluorescent effect. Subjectively, these samples appeared brighter than the untreated and DMEU-treated cottons. The color measurements (UV included) showed slight increases in lightness and redness in the fabric, due to the various whitening treatments. The *b* values indicated marked masking of yellowness by some treatments with measurements ranging from −1.4 to + 4.1 units. Without the fluorescence contribution (UV excluded) the *b* values, which ranged from +4.6 to +6.7, indicated considerable yellowness in the samples, with many samples yellower than the DMEU-treated control (+5.1).

Blue reflectance values of these samples were all higher with ultraviolet included and ranged from 81.6 to 87.4% (untreated 81.4%). With ultraviolet excluded, blue reflectance of many samples was slightly lower than that of the untreated and of the DMEU control.

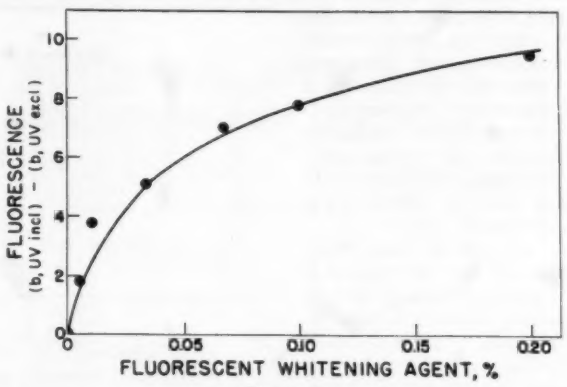

Figure 1
Fluorescence of cotton samples treated with DMEU and various concentrations of fluorescent whitening agent A applied from the same pad bath

The fluorescence of the samples was estimated by the difference between the *b* measurement with ultraviolet included and the *b* measurement with ultraviolet excluded. This simplified method of estimating the effect of fluorescence has been used by Furry and coworkers (*10*). The values obtained ranged from 0.3 for the untreated cotton to 6.8 for the sample treated with DMEU and whitener A by process IV. The whitening effect produced by the various treatments also can be estimated from the blue reflectance measurements by similar calculations.

Of the three methods of processing using DMEU, that in which the fluorescent whitener was applied in a second step to fabric finished with DMEU (III) appeared to give the

TABLE II
Whitening effects of cotton print cloth treated with DMEU and various fluorescent whitening agents

Treatment		Color measurements							Blue reflectance	
		UV included			UV excluded					
Process	Whitener used	Rd	a	b	Rd	a	b	Fluorescence[3]	UV included	UV excluded
—	—[1]	89.6	−0.1	+4.2	89.5	−0.1	+4.5	0.3	81.4	81.2
IV[2]	—	89.8	−0.2	+4.8	89.7	0.0	+5.1	0.3	80.8	80.5
I	A	90.1	+2.1	−1.4	89.5	−0.2	+4.9	6.3	87.4	81.3
II	A	90.4	+2.1	−0.9	89.7	−0.2	+5.2	6.1	86.6	80.9
III	A	90.3	+0.6	+3.0	89.9	+0.1	+5.0	2.0	82.6	80.9
IV	A	90.2	+2.2	−1.3	89.7	−0.3	+5.5	6.8	87.2	80.7
I	B	90.8	+1.8	−0.6	90.3	−0.2	+4.8	5.4	87.3	81.9
II	B	90.2	+1.3	+0.8	89.7	−0.2	+5.1	4.3	84.8	80.6
III	B	90.7	+0.7	+2.9	90.6	−0.1	+4.9	2.0	83.1	81.2
IV	B	90.1	+1.6	+0.2	89.8	−0.3	+5.3	5.1	85.6	80.8
I	C	90.7	+1.8	−0.6	90.5	−0.2	+4.7	5.3	86.8	81.9
II	C	90.6	+1.6	+0.4	90.5	−0.3	+5.0	4.6	85.6	81.0
III	C	89.8	+0.4	+3.4	89.8	−0.2	+4.9	1.5	82.2	80.7
IV	C	90.2	+1.8	−0.1	89.8	−0.3	+5.4	5.5	86.0	80.6
I	D	90.5	+0.2	+3.8	90.4	−0.1	+4.6	0.8	82.7	81.6
II	D	90.6	+1.4	+0.5	90.3	−0.5	+5.8	5.3	87.0	81.3
III	D	90.7	+0.4	+3.5	90.7	−0.2	+4.9	1.4	82.8	81.3
IV	D	90.7	+1.4	+0.7	90.6	−0.5	+5.9	5.2	87.3	81.4
I	E	90.9	+0.9	+1.2	90.2	−0.3	+5.2	4.0	87.0	82.0
II	E	90.6	+0.9	+1.6	90.2	−0.4	+5.8	4.2	85.8	81.1
III	E	90.1	+0.2	+4.1	90.1	−0.2	+5.3	1.2	81.6	80.6
IV	E	89.9	+0.8	+1.9	89.6	−0.4	+5.6	3.7	84.8	80.5
I	F	89.9	+0.3	+3.5	89.8	+0.1	+4.7	1.2	82.4	81.2
II	F	90.1	+0.5	+2.7	90.0	−0.2	+5.2	2.5	84.1	80.8
III	F	90.3	+0.3	+3.8	90.2	−0.1	+5.0	1.2	82.0	80.9
IV	F	90.2	+0.4	+3.1	89.8	−0.2	+5.4	2.3	83.6	80.6
I	G	90.5	+1.5	−0.2	90.0	−0.3	+4.9	5.1	87.2	81.8
II	G	90.8	+1.0	+1.4	90.2	−0.4	+5.7	4.3	86.0	81.1
III	G	90.5	+0.3	+4.0	90.2	−0.3	+5.7	1.7	82.3	80.7
IV	G	90.6	+0.6	+2.7	90.0	−0.7	+6.7	4.0	84.7	80.1
I	H	90.4	+1.1	+1.3	90.0	−0.1	+4.8	3.5	85.4	81.5
II	H	90.7	+0.7	+2.5	90.5	−0.2	+5.3	2.8	83.5	80.7
III	H	90.1	+0.6	+3.0	89.8	−0.2	+5.3	2.3	83.0	80.5
IV	H	89.9	+0.6	+3.3	89.6	−0.3	+5.8	2.5	82.8	79.9

[1] Untreated cotton print cloth (desized, caustic-boiled, and bleached).
[2] Control treatment with DMEU; no whitening agent used.
[3] Fluorescence is the difference between *b* measurement, UV included, and *b*, UV excluded.

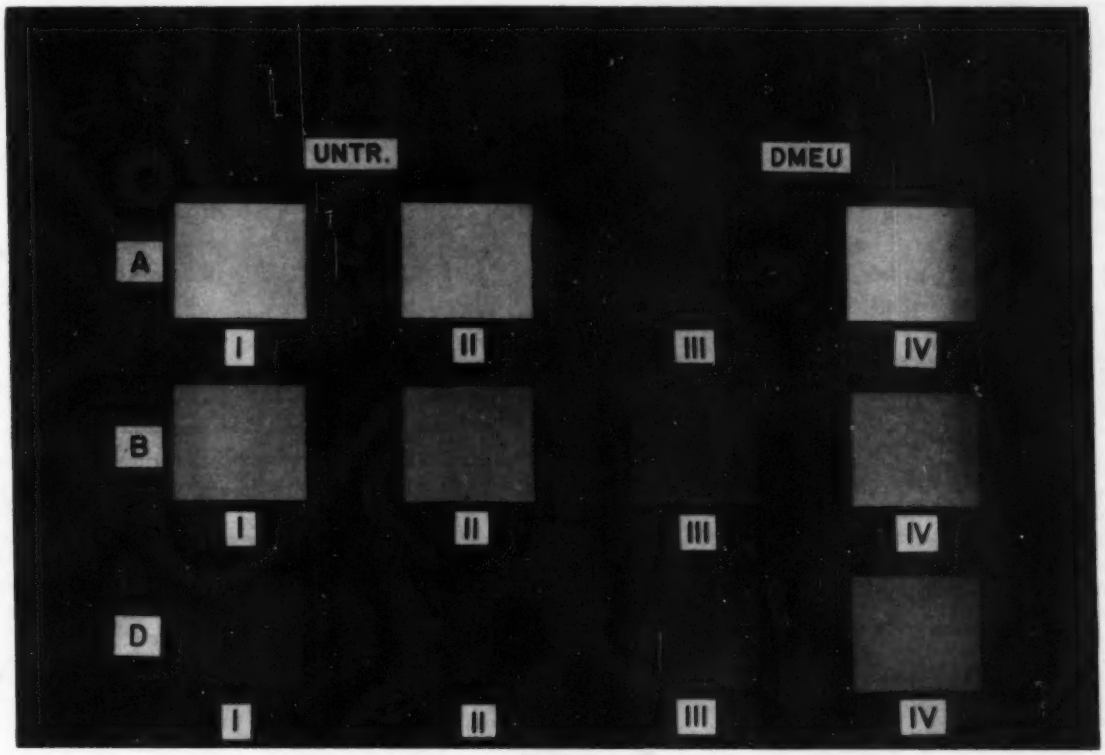

Figure 2

Appearance under ultraviolet radiation of fabrics treated with DMEU and selected fluorescent whitening agents. The letters to the left of the row of swatches indicate the whitener used and the numerals below the swatches indicate the finishing process employed.

poorest results. The other two processing techniques, application of DMEU to fabric treated with whitener (II) and application of DMEU and whitener from the same pad bath (IV), gave results which were essentially equal and which were comparable to treatment of cotton with whitener alone (I).

Technique IV, of course, would eliminate one processing step and is more attractive economically.

CHANGE OF CONCENTRATION OF WHITENER———Using process IV, samples were prepared by treating fabric with baths containing 7% DMEU and 0-0.2% fluorescent whitener A. Figure 1 shows a plot of the fluorescence vs the concentration of fluorescent whitening agent in the pad bath. Fluorescence increased rapidly with concentration of whitener to about 0.066% above which increased concentrations of whitener produced less additional effect. A concentration of 0.1% had been arbitrarily used in the work reported in Table II and was also used in subsequent experiments. It has been shown and discussed by Allen (23) that fluorescence reaches a limit with high concentrations of whitener because almost all the available ultraviolet energy used for exciting the fluorescence is absorbed at high concentrations, and additional whitener cannot absorb any more.

SEMIPILOT-PLANT TREATMENTS———Three of the whiteners were selected for further study and were applied to cotton print cloth on a semipilot-plant scale. The whiteners selected were A, B, and D. Whiteners A and B had produced results among the best in the experiments reported in Table II. Whitener D was selected to include a whitener of another class, and because of possible substantivity to

TABLE III

Whitening effects of cotton print cloth treated on a semipilot-plant scale with DMEU and selected fluorescent whitening agents

Treatment		Color measurements							Blue reflectance	
		UV included			UV excluded					
Process	Whitener used	Rd	a	b	Rd	a	b	Fluorescence[3]	UV included	UV excluded
—	—[1]	89.0	+0.1	+4.7	89.0	+0.2	+5.1	0.4	79.9	79.9
IV[2]	—	87.1	−0.1	+5.9	87.1	+0.1	+6.3	0.4	77.3	77.3
I	A	90.8	+2.3	−1.5	90.1	+0.2	+5.3	6.8	87.6	80.9
II	A	90.5	+1.9	+0.1	90.2	+0.1	+5.9	5.8	85.4	79.9
III	A	88.7	+0.6	+4.0	88.7	+0.2	+5.8	1.8	80.1	78.6
IV	A	89.6	+2.0	+0.5	89.3	+1.4	+6.4	5.9	84.6	78.8
I	B	91.3	+1.7	+0.6	90.7	+0.3	+5.3	4.7	85.7	81.1
II	B	90.5	+0.9	+2.4	90.3	+0.1	+5.9	3.5	83.5	79.9
III	B	87.7	+0.4	+4.8	87.7	+0.2	+6.1	1.3	78.6	77.4
IV	B	90.5	+1.1	+2.8	90.4	−0.1	+6.2	3.4	83.0	79.7
I	D	90.0	+0.5	+3.6	89.6	+0.1	+5.1	1.5	82.1	80.3
II	D	89.3	+0.4	+4.5	89.0	+0.1	+6.0	1.5	80.5	79.0
III	D	88.5	+0.3	+5.0	88.6	+0.1	+6.0	1.0	79.2	78.3
IV	D	91.1	+0.6	+3.5	90.6	−0.2	+6.6	3.1	84.2	80.2

[1] Untreated cotton print cloth (desized, caustic-boiled, and bleached).
[2] Control treatment with DMEU; no whitening agent used.
[3] Fluorescence is the difference between *b* measurement, UV included, and *b*, UV excluded.

TABLE IV

Fluorescence of samples after repeated laundering in a household-type automatic washer

Treatment			Fluorescence[1]															
			After laundering with detergent containing no whitener				After laundering with detergent containing no whitener plus bleach				After laundering with detergent containing whitener				After laundering with detergent containing whitener plus bleach			
Process	Whitener used	Originally	1X	5X	10X	20X	1X	5X	10X	20X	1X	5X	10X	20X	1X	5X	10X	20X
—	—[3]	0.4	(1.7)[3]	(1.5)[3]	(1.5)[3]	(1.5)[3]	(0.8)[3]	(0.6)[3]	(0.5)[3]	(0.4)[3]	3.4	5.4	6.4	7.1	1.4	2.2	2.3	2.0
IV[4]	—	0.4	0.4	0.1	0.4	0.3	0.2	0.2	0.2	0.3	1.1	1.8	2.4	2.8	0.5	0.6	0.9	0.9
I	A	6.8	5.6	4.4	3.9	3.5	5.6	3.5	2.8	1.8	6.2	6.3	6.8	7.2	5.6	4.1	3.1	2.7
II	A	5.8	5.5	5.4	5.4	5.4	5.1	3.9	3.9	2.5	5.7	5.5	5.8	5.8	4.7	3.9	3.2	2.5
III	A	1.8	0.8	0.4	0.4	0.4	0.8	0.5	0.4	0.4	1.4	1.7	2.0	2.9	0.9	0.8	0.7	0.9
IV	A	5.9	6.1	6.1	6.2	5.9	6.0	4.9	4.1	2.6	6.8	6.8	6.6	6.6	5.4	4.4	3.5	2.5
I	B	4.7	3.8	3.1	2.6	2.5	3.8	2.6	2.1	1.2	4.5	5.9	6.3	6.7	3.7	3.3	2.7	2.6
II	B	3.5	3.2	3.0	2.9	2.8	2.9	2.2	2.0	1.3	3.1	3.4	3.7	4.0	2.8	2.2	2.0	1.6
III	B	1.3	0.8	0.6	0.6	0.5	0.9	0.4	0.3	0.4	1.3	1.9	2.2	2.6	1.0	0.9	0.9	0.8
IV	B	3.4	3.5	3.3	3.2	3.1	2.9	2.5	2.1	1.5	3.5	3.8	3.6	3.7	2.9	2.2	1.8	1.5
I	D	1.5	1.6	1.3	1.5	1.4	0.9	0.5	0.5	0.4	2.9	5.5	6.1	6.8	0.9	1.9	2.1	2.2
II	D	1.5	1.2	1.1	1.1	1.2	0.9	0.5	0.5	0.4	1.6	2.2	2.6	3.0	0.7	0.7	0.8	0.9
III	D	1.0	0.6	0.3	0.2	0.8	0.3	0.4	0.3	0.3	1.1	1.6	2.0	2.6	0.4	0.6	0.7	1.0
IV	D	3.1	2.7	2.3	2.3	2.0	1.9	1.2	0.9	0.6	2.6	2.9	2.9	3.1	1.6	1.3	1.2	1.1

[1] Fluorescence is the difference between *b* measurement, UV included, and *b*, UV excluded.
[2] Untreated cotton print cloth (desized, caustic-boiled, and bleached).
[3] Because all samples were washed together in these laundering tests, untreated samples increased in fluorescence by picking up whitener leached from other samples. The figures are presented to indicate the extent of such pickup.
[4] Control treatment with DMEU; no whitening agent used.

DMEU-treated cotton since the whitener is reported to be substantive to wool.

In Table III are shown the whitening effects obtained on cotton as a result of the semipilot-plant applications. It will be noted that the results are somewhat different from the corresponding samples in Table II. Possibly the variation is due to differences between laboratory-scale batch treatment and semipilot-plant continuous treatment. In the latter, slightly higher temperatures were encountered which may have caused the slightly higher degree of yellowness which is shown by the *b* values. With this difference, the data of the two tables appear to be comparable. A photograph of these samples under ultraviolet radiation from GE fluorescent BLB type lamps with UV filters are given in Figure 2. Differences in fluorescence under ultraviolet radiation are strikingly illustrated in the photograph.

These samples were divided into a number of portions and were then evaluated after various types of laundering.

LAUNDERING IN HOUSEHOLD WASHER———Detergents containing no fluorescent whitening agents were practically unobtainable commercially (*6*) but special samples were made up for this study by an industrial source. This detergent is otherwise the same built detergent as is packaged with whiteners and used in large quantities by American housewives for home laundering. Approximately 2.5 g/gallon were used in a 14-gallon washer. When hypochlorite bleach was used, the concentration employed was 0.02% available chlorine. Solution to cloth ratio was about 77:1 and the wash temperature was 140°F.

TABLE V

Fluorescence of samples after commerical laundering and other wash procedures

Treatment			Fluorescence[1]				
				After AATCC Wash Tests			
Process	Whitener used	Originally	After five commercial launderings	1X	5X	After soap-soda ash boil	After three sour cycles
—	—[3]	0.4	6.7	(1.2)[3]	(0.9)[3]	(3.6)[3]	(0.8)[3]
IV[4]	—	0.4	2.1	0.2	0.1	0.3	0.5
I	A	6.8	7.0	1.9	1.4	4.1	6.1
II	A	5.8	6.3	4.4	3.7	5.1	5.7
III	A	1.8	1.9	0.3	0.1	0.4	1.1
IV	A	5.9	6.7	5.3	4.2	5.9	6.3
I	B	4.7	7.0	1.6	1.2	4.3	4.1
II	B	3.5	3.9	2.1	1.6	3.1	3.1
III	B	1.3	2.0	0.3	0.2	0.5	1.4
IV	B	3.4	4.1	2.5	1.7	3.2	3.1
I	D	1.5	6.8	1.1	—[5]	3.4	0.6
II	D	1.5	2.4	0.6	0.4	0.7	0.6
III	D	1.0	1.9	0.4	0.1	0.4	0.6
IV	D	3.1	2.9	1.3	0.9	1.6	1.9

[1] Fluorescence is the difference between *b* measurement, UV included, and *b*, UV excluded.
[2] Untreated cotton print cloth (desized, caustic-boiled, and bleached).
[3] Because all samples were washed together in these tests, untreated samples increased in fluorescence by picking up whitener leached from other samples. The figures are presented to indicate the extent of such pickup.
[4] Control treatment with DMEU; no whitening agent used.
[5] Sample lost before evaluation.

The four wash solutions used in the household-type washer were:

1) Detergent containing no fluorescent whitening agent.

2) Detergent containing no fluorescent whitening agent, but with hypochlorite bleach solution added.

3) Detergent containing fluorescent whitening agents.

4) Detergent containing fluorescent whitening agents and with hypochlorite bleach solution added.

Fabric samples were withdrawn after one, five, 10 and 20 wash cycles. The change in fluorescence of the samples is shown in Table IV. All the samples were washed together and it appears that the whitener leached from some samples and was picked up by the untreated sample during laundering with detergent containing no whitener. Apparently the sample treated by process IV-A also picked up some additional fluorescence during these launderings.

The use of chlorine bleach with the detergent containing no whitener dramatically diminished the degree of fluorescence of samples on successive launderings. The quenching effect of the bleach was also evident after laundering with detergent containing fluorescent whiteners. In the latter case, some of the results were only slightly better than when the detergent contained no whitener.

Samples laundered with detergent containing whiteners, but without added bleach generally showed increased fluorescence on successive launderings.

COMMERCIAL LAUNDERING AND OTHER WASH PROCEDURES ———Table V shows the fluorescence of the various fabrics after commercial laundering (five cycles), AATCC wash test (one and five cycles), a one-hour soap-soda ash boil, ar three sour cycles at pH 3.4.

Commercial laundering, which included a fluorescent whitener in the procedure, generally increased the fluorescence of the samples, although in many cases the changes were

TABLE VI
Physical properties of samples treated with crosslinking agents and fluorescent whitening agents

Process	Treatment: Finishing agent	Treatment: Whitener used	Crease-recovery angle, (W) deg	Scorch test % orig str retained	Fade-Ometer test, exposure to cause slight visible change, hr
—[1]	—	—	83	93	>200
IV[2]	DMEU	—	143	93	>200
IV[2]	DMET	—	—	96	—
I	—	A	87	93	24
II	DMEU	A	140	70	24
III	DMEU	A	138	99	24
IV	DMEU	A	137	75	24
II	DMET	A	—	96	—
III	DMET	A	—	95	—
IV	DMET	A	—	92	—
I	—	B	93	86	100
II	DMEU	B	143	83	24
III	DMEU	B	146	101	24
IV	DMEU	B	142	89	24
I	—	D	94	81	100
II	DMEU	D	137	58	100
III	DMEU	D	141	95	100
IV	DMEU	D	141	53	100

[1] Untreated cotton.
[2] Treated with finishing agent only; no whitening agent used.

small. Untreated cotton exhibited the largest increase, but the final fluorescence was equalled by the samples treated by process I (without wrinkle resistance agent) and those treated by processes II-A and IV-A. Samples prepared according to process III had practically the same fluorescence after laundering as that of the laundered DMEU-treated control.

The AATCC wash test, a very severe procedure which includes a 40-minute sudsing at 203-212°F in the wash cycle, significantly decreased the fluorescence of all samples except the untreated control. As in the household laundering, this sample apparently picked up whitener leached from others during washing.

Except for the untreated and the sample treated by process I-D, the fluorescence of all samples was diminished slightly by the soap-soda ash boil. The small amount of the decrease in fluorescence was somewhat surprising when the severity of this treatment is considered—pH 11 at 212°F for one hour.

For the most part results after the sour cycles showed slight loss in fluorescence. Samples treated with fluorescent whitening agent D were most affected by the souring.

TEXTILE PROPERTIES——Chlorine retention and wrinkle resistance of the samples were determined and are listed in Table VI. The crease-recovery angles produced by the treatments which included DMEU were all good. This property seems to be practically unaffected by the small amount of whitener used in the processes.

Results from the scorch test are interesting, inasmuch as some samples treated by processes II and IV show decreased chlorine resistance. This indicates that the whitener (or perhaps the reaction product of the whitener with DMEU) may furnish sites for chlorine retention. The whiteners by themselves were not responsible for the loss of strength on heating. That is, samples scorched in the usual manner for the test, but without chlorination showed little change in strength, retentions being between 95 and 104%. Samples treated with whitener A when applied with or before DMEU, lost 25 to 30% after chlorination and scorching. However, there was little strength loss after chlorination and scorching when whitener A was applied by itself or after resin treatment. In the latter two cases, it is probable that the whitener is removed by the rigorous bleaching and thorough washing in the test and thus does not cause the cloth to retain chlorine. With DMEU, the whitener is retained and picks up chlorine. Whitener D acts similarly to whitener A. Samples treated with whitener B also show some loss except the one prepared by process III. The latter sample exhibited poor fluorescence indicating low pickup of whitener. These effects cannot be explained without exact knowledge of the constitution of each whitener, but it is apparent that varying substituent groups will have different affinities for chlorine and this should be considered in practical applications.

Even though a whitener may pick up small amounts of chlorine, this effect can be overcome by use of a chlorine-resistant finishing agent with alkaline substituents such as triazone. This is illustrated by samples treated by processes II-A, III-A, and IV-A using DMET instead of DMEU, which are included in Table VI for comparison. The greater chlorine resistance or neutralizing capacity imparted by DMET (*24*) was sufficient to maintain good resistance to chlorine damage when used with fluorescent whitening agent A. This whitener caused as much as 30% loss of strength in the scorch test when used with DMEU.

FADE-OMETER AND WEATHER-OMETER TESTS——Samples were tested in the Fade-Ometer and observed periodically for visual changes (Table VI). All the samples treated with whitener A and all those treated with whitener B and DMEU showed slight yellowing, as viewed in room light, after 24-hour exposure. The sample treated with whitener B alone was more resistant to color change than those treated with this whitener and DMEU. This effect may be similar to that observed with samples treated with DMEU and various classes of dyestuffs (*25*). Apparently fabrics treated with whitener D, which is of the aminocoumarin derivative type, were less affected by the conditions of the Fade-Ometer test. Gantz and Bergman, working with stilbene-type whiteners, have reported recently (*8*) that the carbon-arc Fade-Ometer tests represents a particularly severe accelerated test which does not correlate with results obtained with samples exposed to sunlight.

The untreated and DMEU-treated cotton were unaffected after 200 hours of exposure. The appearance of samples was compared qualitatively under ultraviolet radiation. The Fade-Ometer caused a loss of fluroescence which could be observed under ultraviolet radiation before the visual changes in room light became apparent.

Although samples were slightly discolored in the Fade-Ometer test, breaking strengths of those treated with whitener A were not seriously affected by the simulated outdoor weathering test in the Weather-Ometer. Figure 3 shows a plot of the percentage of the original breaking strength retained after 0 to 40 hours exposure for three samples—untreated fabric, fabric treated with DMEU, and one with DMEU and whitener A by process IV. The two last-named samples were approximately equally affected by the test. The strength of the untreated was severly diminished, losing more than 50% of its strength in five hours.

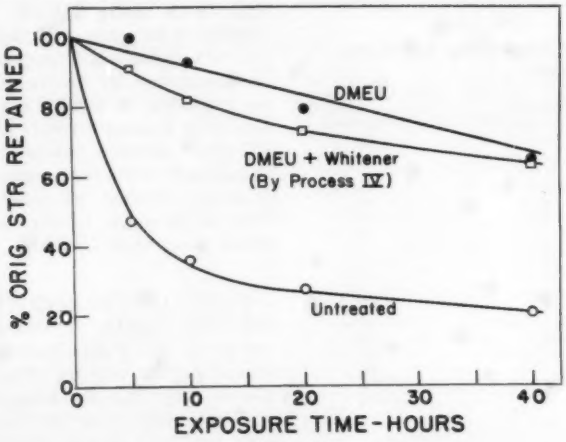

Figure 3
Strength retention of cotton fabrics after exposure in Weather-Ometer

Some fluorescent agents have been reported to cause tendering of cellulose upon exposure to sunlight or carbon-arc radiation (26).

DISCUSSION

For use in "wash-wear" finishing of cotton, three methods of application of fluorescent whitening agents have been considered—application prior to the crosslinking treatment, application after the crosslinking treatment, and application with the crosslinking agent from the same finishing pad bath.

From the data presented, it can be seen that application of the whitener after the crosslinking treatment produced the least whitening effect in the fabric. Generally speaking, crosslinking cotton is not receptive to the fluorescent whitening agent. However, this type of treatment has received some industrial attention. A patent has been issued on a process which involves the use of an oxygen bleaching agent, such as sodium perborate, and a fluorescent whitening agent to improve the whiteness of crosslinked fabrics (27).

Application of a fluorescent whitening agent to cotton prior to the crosslinking treatment is very effective in producing brilliant white "wash-wear" cotton. The optimum application conditions for the whitener may be used without effect on the subsequent "wash-wear" finishing of the fabric. However, this is an added step, and the cost of the whitening operation must be considered by the finisher. Nevertheless, this process is used by many cotton finishing plants.

Application of the whitening agent along with the crosslinking agent from the same pad bath is potentially the most attractive of the three processes studied. With the whiteners used in this investigation, no compatibility problems were encountered. The whitening effect produced was a function of the fluorescent whitening agent used with the stilbene derivative type apparently producing the best results. Durability of the whitening effect, again, was a function of the whitener, but was generally good. Likewise, wrinkle resistance of the fabric was practically unaffected by inclusion of whitener in the treatment. With some combinations of crosslinking agent and whitener, chlorine retention may be a problem. However, if a crosslinking agent with excellent chlorine resistance or high neutralizing capacity is used, such as a triazone derivative, there seems to be no added problem of chlorine damage due to the whitener. Also, the whitener must be carefully chosen to avoid light discoloration, although accelerated tests may not be a reliable tool for determining this property.

Although the above tests were limited in scope, it would appear that the use of fluorescent agents by the finisher is attractive. In fact, these agents should be considered analogus to the usual dyeing processes where the dyeing may precede the wrinkle-resistance treatment or be conducted simultaneously (*28, 29*). Use of a fluorescent agent should produce the brilliant bluish white finish preferred by the consumer and careful selection of the agent should make it durable for a considerable portion of the life of the garment.

SUMMARY AND CONCLUSIONS

Processes for the treatment of cotton fabric for "wash-wear" which include the use of fluorescent whitening agents have been investigated. Treatment with whitening agent prior to and following the crosslinking step and treatment with the crosslinking agent and the fluorescent whitener together in the same pad bath were studied. Finishing was carried out both by laboratory-scale batch treatment and by semipilot-plant continuous treatment. The crosslinking agents employed were dimethylol ethyleneurea and dimethylol ethyltriazone. Eight fluorescent whitening agents, representing the principal types used in current industrial practice, were employed.

In general, the stilbene derivative-type whitener produced the most pronounced brightening effects. Treatment with whitener after crosslinkage was least effective due to the greatly reduced receptivity of the fabric to the whitener. The other two methods of whitener application were about equally effective. However, treatment with whitener and crosslinking agent from the same pad bath apparently would have economic advantages due to the omission of one processing step.

Durability of the fluorescent whitening effects to various home-type launderings, to commercial laundering, and to laboratory washing and souring was determined. Changes observed after these launderings and tests differed with the whitening agent and method of application employed.

The whitening agents had practically no effect on the wrinkle-resistance properties of the finished fabrics. However, chlorine damage to some samples was increased when certain whiteners were used. Apparently the whitener introduced sites for chlorine retention in the finish. This was overcome if a highly chlorine-resistant crosslinking agent or one with good neutralizing capacity was employed.

ACKNOWLEDGMENTS

The authors wish to express their thanks to the following: to E Allen of American Cyanamid Co, R C Allison of Geigy Chemical Corp, E A Wolff of Ciba Co, Inc, and P W Ifland of the Procter and Gamble Co for generously supplying samples and

technical information for this study; to Milton J Hoffman for assistance in the pilot plant; and to members of the Textile Testing group of SURDD for textile property tests.

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11th ADR Award———

(*concluded from page 9*)

Frank J Rizzo was born in Lawrence, Mass, on January 23, 1910, and was educated in public schools in Andover, Mass. He subsequently attended Phillips Academy at Andover, from which he was graduated in June 1927. In the fall of 1927, he transferred to Harvard University from which he received a BS in organic chemistry in 1931.

Following his graduation from college, Mr Rizzo went to work for the now defunct American Woolen Co as its chief analyst for a period of approximately nine months. He subsequently transferred to the Arlington Mills Div, Wm Whitman Co in October 1932, where he went all the way from a laboratory assistant to assistant chief chemist with specific responsibility for research. He left in September 1946 to join the R&D Laboratories, Philadelphia Quartermaster Depot.

In 1953, the Laboratories were transferred to Natick, Mass, and Mr Rizzo was one of the first to make the transfer from Philadelphia to the new Quartermaster R&D Command.

During his years with the Quartermaster Corps, he has been essentially in the same general position he now holds, namely, chief of the Dyeing and Finishing Laboratory, and has been responsible for all of the color aspects of the U S Army uniform. One of the important areas covered has been the establishment of sound standards and tolerances against which all procurements of textiles for the U S Army are based as complements to the written specification.

Mr Rizzo has been involved in the development of the colors for the U S Air Force Uniform, the U S Army uniform, particularly the Army greens and Army blues, and has also undertaken work for the Marine Corps and U S Navy.

He has had a number of papers published in technical journals, particularly in the *American Dyestuff Reporter*, and a sizable number of Textile Series Reports on both U S Army materials as well as those of other Services. He also holds a number of patents in the general field of dyeing and finishing.

In addition to his AATCC activities, Mr Rizzo holds membership in the American Chemical Society, Optical Society of America, Inter-Society Color Council, Research Society of America, and Association of Harvard Chemists. He has been an active member of a large number of AATCC technical committees, including membership on the Technical Committee on Research, and has served as chairman of at least one committee, the Committee on Weathering. He served as a member of the Council from 1956 to 1959, was vice chairman of the Northern New England Section from 1945 to 1946, and is presently vice chairman of the Section for a second time.

He has been chairman and a member of several Intersectional Technical Paper Competition Committees for both the Northern New England and Delaware Valley Sections. He was a member of the Study Committee for the Intersectional Contest, and has represented AATCC as an official delegate to the International Organization for Standardization.

Mr Rizzo, who resides at 23 Travis Rd, Natick, Mass, is married to the former Mary Widdop of Lawrence, Mass. They have two children.

Alvin O Ramsley, who is chief of the Color Properties Research Section, Textile Dyeing Laboratory Branch, at the QM R&E Center at Natick, received his BS degree from Houghton College in 1943 and his AM degree from Columbia University in 1948. He is a member of the Optical Society of America, American Chemical Society, Research Society of America, and the American Scientific Affiliation.

Previous ADR Award winners are shown in the following table.

Year	*Authors*	*Title*
1951	Mason Hayek and Fred C Chromey, E I du Pont de Nemours & Co, Inc	"The Measurement of Static Electricity"
1952	Charles J Lyle, Joseph J Iannarone and Robert J Thomas, E I du Pont de Nemours & Co, Inc	"High-temperature Dyeing of New Synthetic Fibers"
1953	Robert H Blaker, E I du Pont de Nemours & Co, Inc	"Principles of Copper Dyeing of Polyacrylonitrile Fibers: The Controlled Redox-Potential Method"
1954	Charles L Zimmerman, American Cyanamid Co	"The Application of Dyes to Textile Fibers at High Temperatures"
1955	Henry E Millson, American Cyanamid Co	"The Microdyeoscope: Microscopical Observations of Dyeing"
1956	Fred Fortess, Celanese Corp of America	"Dyeing, Finishing and Heat Treating Arnel Triacetate"
1957	Irving A Berstein, Controls for Radiation, Inc	"Applications of Radio-Activity in the textile Industry"
1958	Emery I Valko, Giuliana S Tesoro and Waldek L Ginilewicz, Onyx Oil and Chemical Co	"Elimination of Static Electricity from Textiles by Chemical Finishing"
1959	Charles R Williams, Monsanto Chemical Co	"Quantitative Measurement of Fabric Appearance—Practical Evaluation of 'Wash-and-Wear' Characteristics"
1960	Victor S Salvin, Ruth A Walker, and John R Adams Jr, Celanese Fibers Co	"Relation of Dye Structure to Propertes of Disperse Dyes"

Palmetto Section———

LABORATORY EVALUATION METHODS FOR FIBER LUBRICANTS*

H T BUCKLEY
Emery Industries, Inc
Cincinnati, Ohio

INTRODUCTION

IN the past, the fiber processor has often relied upon the data, advice, or persuasion of the lubricant supplier in attempts to up-grade the performance of his lubricant program. Today, however, his interests in bench screening prior to extensive and expensive mill testing have increased with his increasing complexities of new fibers, blends, and modernized equipment and speeds.

As one of the suppliers of fiber lubricants, we have frequently been asked for suggested lab tests that could directly or indirectly relate to mill performance. Quite naturally the interests of mill personnel have usually centered upon screening tests that are simple, rapid, and not requiring expensive lab equipment.

Toward this end, we would like to discuss several test methods that might be considered for this purpose, classifying them for simplification into

1) physical properties,
2) performance characteristics, and
3) analytical procedures.

PHYSICAL TESTS

SOLUBILITY AND STABILITY ———Since fiber lubricants are usually applied from a water-phase system, their degree of solubility and stability when mixed with water can have a bearing on their overall performance.

The nature of its water miscibility will permit you to visually differentiate from a completely water-soluble type to a very coarse particle size distribution of the lubricant in the water system.

Furthermore, this water-lubricant system must possess sufficient stability characteristics to assure uniform application to the fiber material.

Figure 1 illustrates three general types of water solubility, one being completely water-soluble, another dispersible, and the third an emulsion. All three are extremely stable and show no change in appearance after prolonged storage.

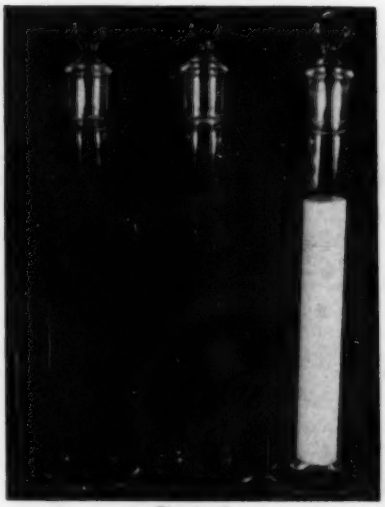

Figure 1

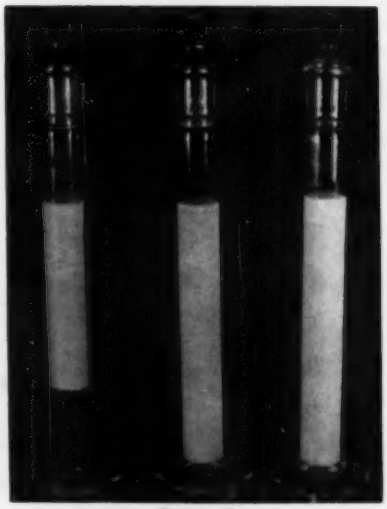

Figure 2

Figure 2 illustrates three general types of instability. One shows a large water layer at the bottom of the cylinder, another shows a small oil layer at the top, and the third emulsion has changed into a two-phase system with the coarser particle sizes in the upper layer.

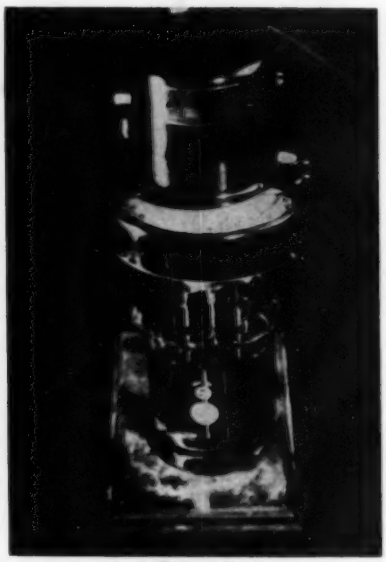

Figure 3

VISCOSITY MEASUREMENTS——Measuring the viscosity of a lubricant will assist in predicting its handling properties in mill use. It might also define whether or not the lubricant could be applied neat if so desired. In addition, it will help in classifying the lubricant, such as a "thin" type, which is often used on greasy or reworked stock, or a "heavy-body" type, which is frequently used on slippery-type fibers.

Figure 3 shows a Brookfield Viscometer, which is highly recommended for its simple and rapid, direct reading features covering an extended range of dynamic viscosities.

Emulsion viscosities with varying amounts of water-to-lubricant ratios can also provide interesting information as an aid in predicting uniformity of the emulsion application.

*Presented before the Palmetto Section, April 15, 1961 at the Richmond Hotel, Augusta, Ga.

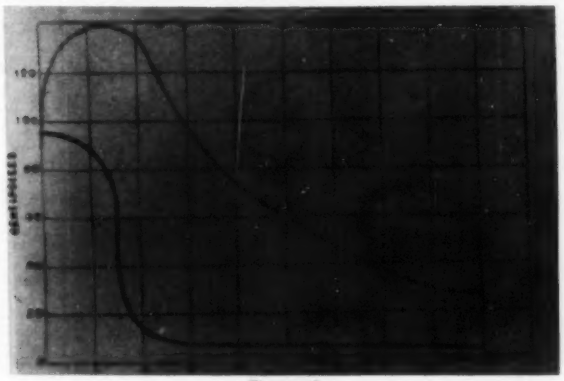

Figure 4
Emulsion viscosities—75°F

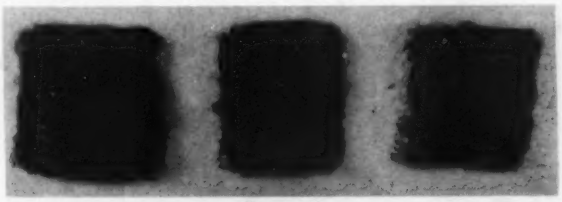

Figure 5
Rusting test

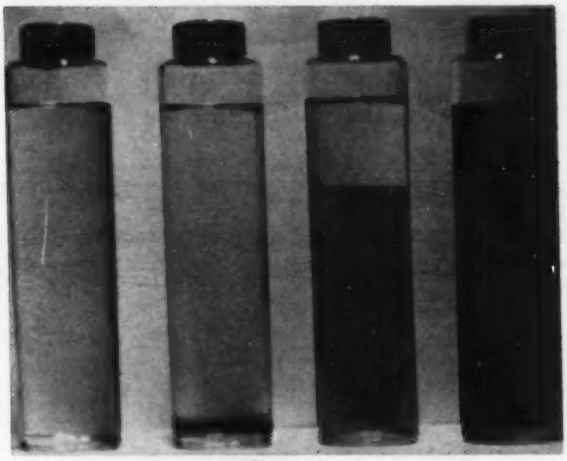

Figure 6
Warm and cold storage test

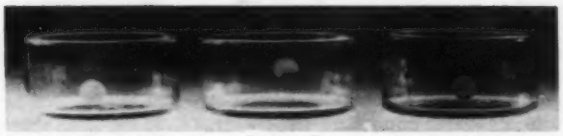

Figure 7

Figure 4 shows two commercial lubricants that have quite noticeable differences in emulsion-viscosity characteristics. One lubricant drops very sharply in viscosity as water is added to it and quickly levels out in emulsion viscosity. The second lubricant increases in viscosity at the beginning of water addition and then slowly decreases in viscosity. Obviously, small changes in water-to-lubricant ratios in the conventional ranges make a negligible difference in emulsion viscosity of the first lubricant and a pronounced difference in the viscosity of the second lubricant emulsion, which in turn can affect uniformity of lubricant application.

RUST INHIBITION———A lubricant should not be corrosive or sensitive to the common metals encountered in the trade and should possess rust-inhibiting properties in water-emulsion form for suitable protection of nonstainless-steel machinery.

Figure 5 illustrates a simple test in evaluating lubricants and their water emulsions for rust-inhibiting properties. Ordinary steel-wool pads have been exposed to 10% solutions of the lubricants in closed jars, and the appearance of the steel wool pads noted after one week's storage.

One lubricant completely protects the large surface area of the steel wool pad from rusting, a second shows localized pitting effects, while the third demonstrates no rust inhibiting properties as seen by the large amount of rusting that developed.

STORAGE STABILITY ——— A lubricant must be stable to temperature variations that normally occur in the transportation and storage of the material.

A warm storage temperature of 120°F is considered maximum that might be experienced in boxcar or warehouse storage, and a temperature of 40°F is arbitrarily chosen as typical of a cold warehouse. Storage tests in a still oven at 120°F and in a commercial refrigerator at 40°F for 24-hour intervals are therefore suggested for checking stabilities of lubricants on storage.

Figure 6 shows one lubricant that separates its emulsifier phase at the bottom on warm storage, although it has satisfactory cold-storage stability. A second lubricant shows the opposite effect in that it is stable to warm storage, but it separates a mineral layer at the top on cold storage. These effects are quite typical of lubricants that have either warm or cold storage instabilities.

VOLATILITY———The Wool Associates of the New York Cotton Exchange have included in their worsted lubricant specifications a volatility test that consists of exposing about five grams of lubricant in a weighing dish for 24 hours in a still oven at 105°C. The lubricant should not gain over 5% in weight or lose more than 10% in weight under these conditions.

Figure 7 shows three mineral oils that have been given this volatility test. Not only will the volatility characteristics of oils vary considerably, but the color stabilities can also differ appreciably.

PERFORMANCE TESTS

SPREADING AND PENETRATION———Figure 8 illustrates four commercial lubricants having quite different spreading and penetrating characteristics. Since penetration is largely a rate function, its effect cannot be shown here. However, by coloring a 3:1 water-to-lubricant emulsion and placing a drop of the emulsion on a scoured wool flannel, not only the rate at which the drop

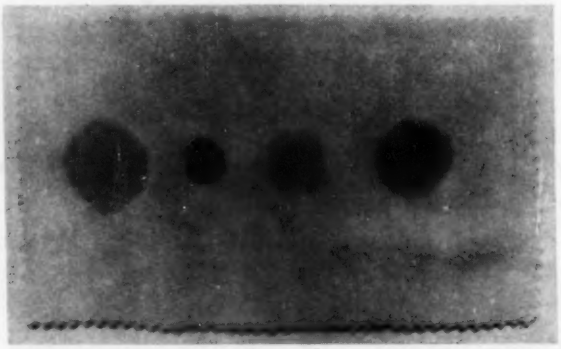

Figure 8
Spreading and penetrating test

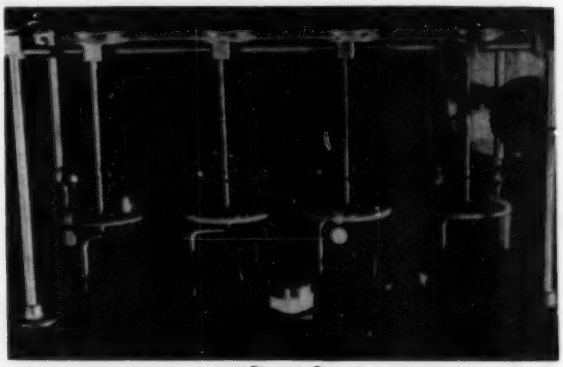

Figure 9

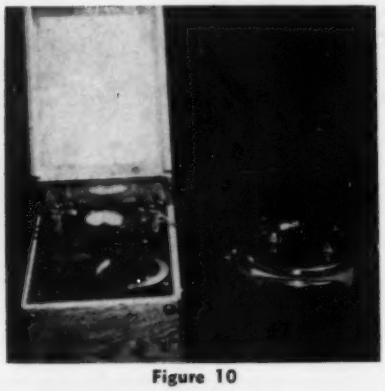

Figure 10

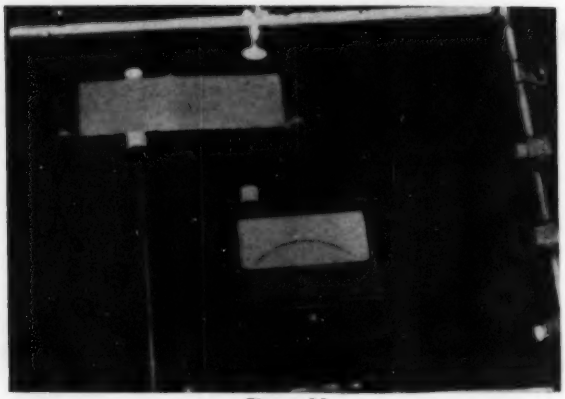

Figure 11

sinks into the cloth but also the nature of its spreading laterally on the cloth can give a qualitative comparison of these important factors.

One lubricant emulsion showed rapid penetration and extensive spreading effects; a second gave rapid penetration but poor spreading; a third gave slow penetration and poor spreading; and the fourth lubricant was rapid in penetration and good in spreading characteristics.

Good spreading and pentrating properties of a lubricant emulsion should provide more uniform application of the water-lubricant system to the fibers and thereby assist in obtaining more uniform performance in processing.

SCOURABILITY TESTS——By carefully limiting the scouring variables, such as time, temperature, concentration of scouring agents, and amount of agitation, differences in the ease of scourability of various lubricants can be shown in the laboratory.

The NAWM specifications of lubricants for worsted top include a scourability test, which is a modification of the AATCC Test Method 1-1957 using a Launder-Ometer. However, we still prefer the older A D Little procedure, modified with mechanical agitation in place of hand stirring, to show greater differences in *ease of removal* between two fiber lubricants.

Figure 9 shows a lab-built scouring machine in which skeins impregnated with lubricants are given a very mild scour and rinse, and ether extraction data determined before and after scouring for percent removability.

For comparative purposes, lubricants that would require only mild mill scouring will show a range of 75 to 95% removal in this A D Little test. Corresponding removals in the NAWM test will fall in the narrow range of 95 to 100%.

Lubricants showing a removal of 50 to 75% range by the A D Little test can be sufficiently removed in mill scouring with conventional scouring conditions.

Lubricants showing less than 50% removal by the A D Little test should be given a more thorough mill scour for effective removal. In this lower class of lubricant scourabilities, the spread between the modified A D Little test and the modified AATCC test becomes even greater. For example, a lubricant showing a 24% removal in the former test gives a 64% removal in the latter test.

STATIC CONTROL TESTS——Figure 10 shows an electrical resistivity apparatus which can be used in evaluating static electricity on textiles, since resistivity bears some relationship to the ability to dissipate static voltage.

Currently, however, a simple rub test is more frequently used in our laboratories in measuring relative static control of various lubricants.

Figure 11 shows the principal parts of the rub test apparatus, where the sample is rubbed on steel, polyethylene, similar or dissimilar fibers, etc, the electrical charge collected on a copper sleeve, and the static volts read directly on an electrostatic voltmeter connected to the copper sleeve. Conditions of testing, such as amount of lubricant, relative humidity, speed, etc, can be varied to obtain suitable but relative comparisons of lubricants for their highly important property of static control.

YELLOWING RESISTANCE TESTS——The Wool Associates of the New York Cotton Exchange include in their specifications a light

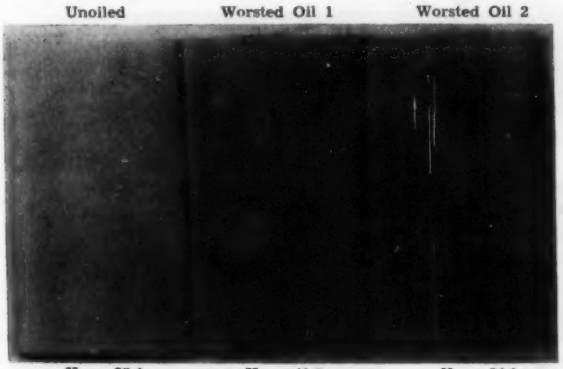

Figure 12

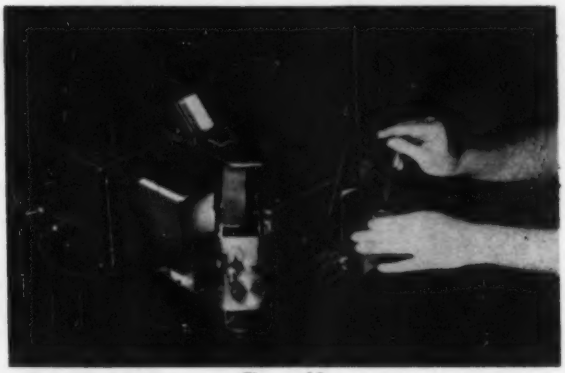

Figure 13

stability test in which a worsted cloth containing 10% lubricant is exposed for 20 hours in the Fade-Ometer. No more than a barely perceptible change in color is defined as necessary to pass this test.

The AATCC Standard Test Method 62-1956 for "Resistance of Wool Oils to Oxidation in Storage" probably gives a more complete picture of the influence of lubricants on the yellowing of stored goods, but it requires the longer period of three weeks oven aging of similarly treated cloth at 120°F.

Figure 12 shows the results of the AATCC test on two well-established worsted lubricants, the one in the center showing noticeably more yellowing than the other lubricant on the right or the unoiled standard on the left.

The numerical data at the bottom of each sample represent yellowness factor units as measured on a Gardner Multipurpose Automatic Reflectometer.

Figure 13 is a picture of a reflectometer in which reflectance values through amber, green, and blue filters are obtained. This instrument is also used in evaluation of degrees of whiteness and greyness, as would be

Figure 14 shows the simple formula for computing the relative degrees of yellowness of the AATCC aged samples.

$$\text{YELLOWING FACTOR} = \frac{\text{Amber Reflectance} - \text{Blue Reflectance}}{\text{Green Reflectance}}$$

Figure 14

Occasionally inquiries are received concerning a quick method of determining the susceptibility of lubricants to auto-oxidation.

Figure 15 shows a Mackey tester which measures the heat produced by decomposition of peroxides formed

Figure 15

Figure 16

largely by oxidation of unsaturated material. Briefly, absorbent cotton saturated with the lubricant under test is packed in a wire cage with a thermometer placed in the center of the packed cotton, heat is then applied to the material by the use of a boiling water jacket, and the temperature change of the oily cotton is plotted. Usually a slow rise in temperature occurs, called the induction period, which is then followed by a rapid increase in temperature when rapid oxidation begins. The time required to reach 105°C is a convenient means of measuring the relative oxidation resistance of samples under test.

LUBRICATION TESTS——Figure 16 illustrates a very simple method of measuring frictional effects.

One end of a lubricated yarn is fastened to a spring scale, passed around a grooved steel wheel, which is driven by a lab-size, constant-speed motor, threaded over a free pulley, and suspended at the other end under small tension. Both fiber-to-metal and fiber-to-fiber frictional comparisons can be made by changing the type of surface of the grooved wheel.

Figure 17 shows a Shirley Yarn Friction Tester as an example of a ready-made instrument for more detailed studies of coefficients of friction.

DRY SOILING RESISTANCE TEST——The specialized use of fiber lubricants for yarns going into floor coverings must be given special consideration, since the lubricant is not usually removed and therefore can influence the soiling properties of the floor-covering material.

The type of fiber, the amount of lubricant, and the nature of the lubricant are general factors that can affect dry soiling resistance.

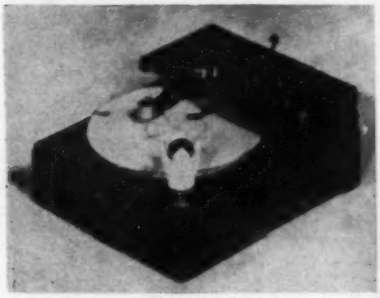

Figure 17

The preferred test in evaluating soiling tendencies is to give the carpet an actual exposure to floor traffic. The disadvantages of this test are the longer time element involved and the possible variation in the traffic conditions.

Several lab tests for soil resistance have been proposed in the literature, and we are currently working on such a test that shows some promise in duplicating floor test results with reasonable reproducibility. In this test a dry soil is applied to cut yarns by Launder-Ometer tumbling and dusting, and a soiling index is then computed from reflectance values obtained before and after the soiling.

Figure 18 shows a selected comparison of two commercial paste lubricants and an oil-type lubricant for dry soil resistance of a viscose carpet yarn. The original, unsoiled yarn is at the left; next is the soiling of the yarn with *no* lubricant on the yarn; the third sample has been treated with a paste lubricant that actually improves its soil resistance; the fourth is another paste lubricant which soils somewhat more than the unlubricated yarn; and the last sample was treated with the oil-type lubricant, which as expected shows the greatest amount of soiling.

ANALYTICAL TESTS

Probably the most frequently encountered questions on analytical test methods that have been brought to our attention involve extraction procedures, and determinations of moisture and mineral oil contents of the lubricants.

Figure 19 shows a conventional Soxhlet battery for determinations of oil, grease, or lubricant content. The standard ASTM method D-404-44 of ethyl ether extracting and ethyl alcohol extracting is normally used, adding the extracted matter weights to the bone-dry weight of the sample for computing percent extracted matter.

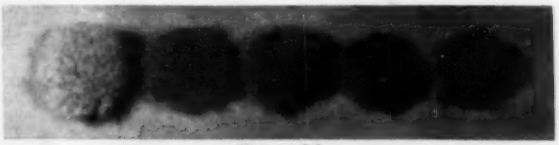

Figure 18

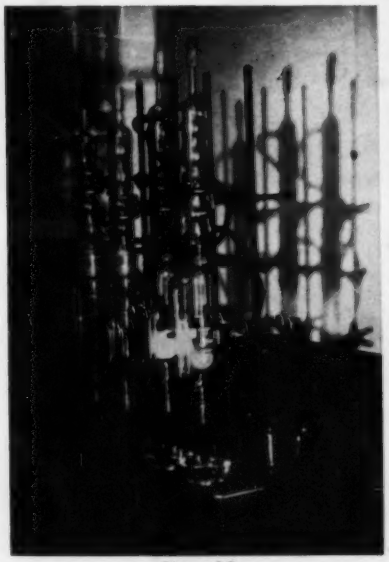

Figure19

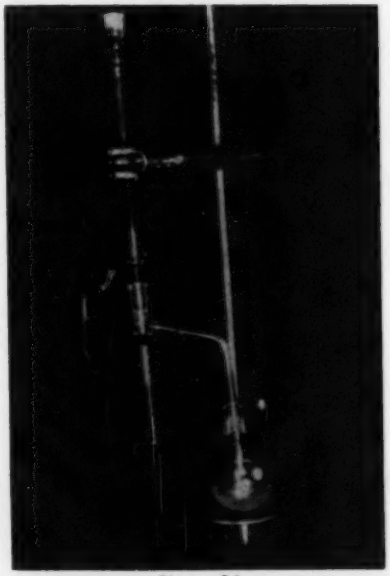

Figure 20

Figure 20 is a picture of laboratory apparatus for determining moisture in a lubricant. The procedure involves a simple azeotropic distillation of the moisture from anhydrous xylene or Stoddard Solvent, collecting the moisture in a graduated trap, and reading the volume obtained directly on the trap.

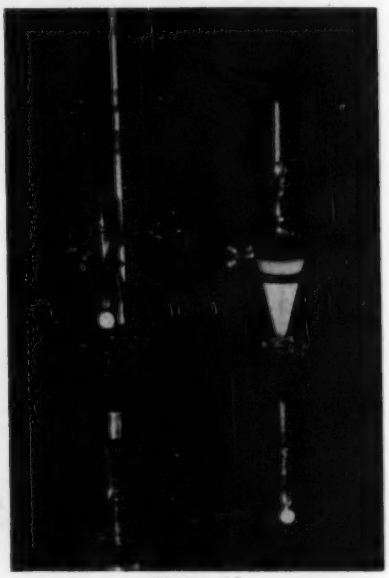

Figure 21

Figure 21 shows the two principle steps in determining the unsaponifiable matter content of a lubricant. In the mineral-type lubricant blends, this unsaponifiable matter is largely the mineral-oil phase of the lubricant. The first step involves the boiling of the sample in alcoholic KOH, and the second step consists of separating the unsaponifiable portion in the petroleum ether layer.

SUMMARY

In summary, several simple lab tests have been briefly reviewed which might be helpful to a fiber processor in predicting the mill performance of a given lubricant.

Obviously, the standardization of such test methods for the whole fiber processing field is a difficult objective to consider, but certainly any step toward this end would appear to be in the right direction.

Consequently, we openly invite and encourage suggestions on improving these tests for the evaluation of fiber lubricants. Only through such cooperative effort might we expect to further simplify where needed, or expand on certain test procedures where it might be helpful, and thus be able to improve upon such a bench screening program for fiber lubricants.

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Oct 26-27 (Sheraton Hotel, Philadelphia, Pa); TCR, research and administrative committees
Nov 17 (Hotel Sherman, Chicago, Ill); Council and Annual Meeting (2 PM)
Jan 26 (Hotel Commodore, New York, NY); Council (10 AM)
Apr 12-13 (Francis Marion Hotel, Charleston, SC); Council, administrative and research committees

NATIONAL CONVENTIONS
Nov 14-17, 1962 (Atlanta Biltmore Hotel, Atlanta, Ga); Oct 30-Nov 2, 1963 (Boston, Mass); Oct 1-3, 1964 (Hotel Statler, New York, NY); Oct 14-16, 1965 (Conrad Hilton Hotel, Chicago, Ill); 1966 (Philadelphia, Pa)

DELAWARE VALLEY SECTION
Oct 27, Dec 1 (Beck's Restaurant, Philadelphia, Pa)

HUDSON-MOHAWK SECTION
Oct 13 (Albany—General Aniline Tour); Nov 17 (Albany—Nopco, Fiber Lubricants); Jan 12(Amsterdam—Monforts Reactor and the Jet Dyeing Machine); Mar 9 (Amsterdam—Water Repellents, 3M); May 25 (Albany—Historical Aspects of Dyeing); June 22 (Amsterdam—Annual Outing)

METROPOLITAN SECTION
Oct 20 (Kohler's Swiss Chalet, Rochelle Park, NJ—"Cellulosic Reactants", panel); Nov 17 (Suburban Golf Club, Union, NJ—Plant visit to Shell Chemical Co Laboratories); Jan 19 (New York, NY—"Costs", D M Lewis, Colonial Piece Dye Works); Feb 16 (New York, NY); April 13 (Ladies Night—Margaret Ingersol, *Vogue*); May 18 (Kohler's Swiss Chalet, Rochelle Park, NJ—"Nonwovens—Dyeing and Finishing", panel); June (Outing)

MID-WEST SECTION
Nov 18 (Chicago, Ill)

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Oct 20 (Quartermaster R & E Command, Natick, Mass); Dec 18 (Motel 128, Dedham, Mass); Jan 19 (Woodlawn Country Club, Newton, Mass)

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Oct 7-8 (Annual Outing, Rancho Santa Fe, Calif;) Nov 17

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Jan 20-21 (Poinsett Hotel, Greenville, SC); April 14-15, 1962 (Francis Marion Hotel, Charleston, SC)

PIEDMONT SECTION
Jan 27 (Hotel Charlotte, Charlotte, NC); Apr 7 (Hotel Barringer, Charlotte, NC); June 8-9 (1962 Outing—Mayview Manor, Blowing Rock, NC); Sept 29, 1962 (Hotel Barringer, Charlotte, NC)

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AATCC's exhibit booth at the National Chemical Exposition, held in conjunction with the 140th meeting of the American Chemical Society, in Chicago, Ill, September 5–8. L to r: Roy E Martin, Swift & Co Research Laboratories, ACS chairman of the Special Exhibits Committee; George J Mandikos, technical secretary of AATCC; Miss L Schaller, Institute of Gas Technology; and George T Goforth, Office of Civil Defense.

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Papin and His New Digester———

(concluded from page 28)

During Papin's lifetime and for almost a century afterwards, the bleaching of linen and cotton was a long, drawn-out series of operations. The process consisted of alternate immersions of cloth in alkali and then long exposures to the sun and air. This took many months. (13). If Papin had been familiar in detail with the problems of bleaching, he might well have conceived the idea of trying his pressure vessel for improving the alkaline treatment of linen and cotton. This would have furnished a partially bleached fabric which still would have required long exposures to the sun for a full white.

There are no records of any bleacher making use of Papin's ideas until about the year 1800. At this time the great French chemist, Chaptal, and the Englishman, Turnbull, both published material concerning the use of apparatus for the alkaline treatment of textiles under elevated temperatures. Turnbull's process seems an exact copy of Chaptal's.

Chaptal realized that he was using Papin's principle in his process for in discussing the apparatus, he wrote—

> "We have been acquainted for many years with the process by which the natives of the east bleach their cotton: I practiced this process at Montpellier with the happiest success; and it is the only one at present known: we may therefore regard it as constant in its effects, and advantageous in its results. The apparatus consists of an old boiler about six feet high by five in diameter. The bottom of this boiler is of copper and the sides are of the same metal, to the height of 18 inches: the rest of it is of good hewn stone. The upper aperture of the boiler is 18 inches in diameter, and it is closed with a round copper stone cover. Upon the edges of the boiler which forms the base of this kind of Papin's digester, we place a frame or grating made of pieces of wood, which leave a small interval between them. Under this oval boiler is the fire of the furnace intended to give the necessary heat to the operation." (14).

Another question arises. Why did not Chaptal use the type of kier shown by Papin in 1681? After all, this was the basic form which came to be used in the middle of the last century and is still used today. Perhaps the explanation lies in the fact that while Papin's "kier" was the most suitable, the facilities for manufacturing large pieces of metal equipment of such design were not available during Chaptal's time. It was not until about 1825 that large metal vessels could be fabricated to withstand pressure and at this time the first true Papin kiers were put into use.

In the matter of pressure dyeing, Papin was simply about two-and-a-half centuries ahead of his time. Pressure dyeing has been needed only within the last decade and mainly because of the advent of some of the new synthetic fibers difficult to dye under ordinary conditions.

There is no important moral to our story. It is obvious that Papin heretofore has not been properly honored for his contributions to the textile industry of his inventions of the pressure kier and pressure dyeing. Once again those of us working in the textile wet processing industry must realize how much we owe to many well-known and obscure figures of the past working in fields which often seem far removed from our own. But then, who could have guessed that the little supper described by Evelyn in 1684 could conjure up the image of large textile bleacheries processing millions of yards of cotton cloth in thousands of Papin's digesters two centuries later?

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(12) Weld, "A History of the Royal Society," Vol I, p 380-1.
(13) Edelstein, Sidney, "A Frenchman Named O'Reilly," *Am Dyestuff Reptr* 47, #8, 253-4 (April 21, 1958).
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Abstracts———

(continued from page 24)

Laboratory evaluation techniques for stabilizers should simulate the conditions under which the compounds will be processed and used. The authors describe several heat stability tests, in which the maxmum temperature is around 180°C. To test light stability it is customary to use u v lamps to accelerate the effect of sunlight.

Ten references to the literature are cited.

●

Procion Fiber-reactive Dyes for Cotton and Rayon Knitted Goods

Lunn, K A, *Dyer* 125, 341-2, Mar 10, 1961

"When one considers that Procion dyes have been commercially available for less than five years, the impact they have had on the textile industry has been without precedent in the field of dye technology."

As a range, the Procion normal (or cold-dyeing) brands are generally preferred to the Procion H or "hot" types for beck dyeing. The former are more reactive, require less salt and alkali for exhaustion and fixation, and can be applied at room temperature. Both types, however, produce shades of high wet and light fastness. Intermixing of the two types is not recommended and, indeed, is not necessary in batchwise dyeing.

The author gives instructions for beck-dyeing of the cold-dyeing Procions on cotton knitwear at room temperature, using soda ash to effect fixation of the dyes. Several typical formulations are described.

In comparing Procion dyes with the conventional types of dyes normally used hitherto for dyeing cotton and viscose rayon knitted goods, the following points should be considered, he says: 1) simplicity of application; 2) better fastness; 3) broader shade range; 4) shorter time of dyeing; 5) lower overall cost; 6) superior quality of the resulting dyeings.

Compared with diazotized and developed dyes, the Procions have better wet fastness and usually superior light fastness, together with a better range of shades and a much shorter dyeing operation (one bath instead of three).

Compared with azoic dyes, the Procions produce

more level dyeings, especially on tubular knitted goods, and are faster to rubbing. The available range of shades is also much greater.

Compared with vat dyes, the Procions are more level-dyeing (no difficulty with premature oxidation), and offer some shades, especially bright reds and blues, that are not available in vats.

Compared with direct dyes, the Procions have greatly superior wet fastness.

The author also refers briefly to the "pad-batch" method of applying the Procions, a recent development.

●

Comparative Wool Scouring Studies. 1. Yield and Regain Differences between Solvent and Aqueous Methods

Williams, V A, *Textile Research J 31, 472-7, May 1961*

Mill trials, comparing solvent degreasing with soap-soda scouring, have indicated a higher total yield of wool obtained from solvent degreasing. Although there has been no previous comparative yield study, the effect of soap-soda scouring on wool has been examined by various workers. It is known that wool may be damaged during soap-soda scouring by excessive alkaline conditions or too great temperature. However, it would appear that, provided temperature does not greatly exceed 50°C and excessive use of alkali is avoided, soap-soda scouring does not cause significant damage.

The present paper describes laboratory experiments designed to obtain further information on the apparent differences in yield observed in the above mill trials.

Laboratory experiments were carried out on different types of fleece, which were scoured by the following methods:

1) Solvent scouring, using white spirit.

2) Simulated soap-soda scouring, in which the soap was replaced by a nonionic detergent.

3) Alkaline nonionic detergent scouring, using soda ash in the first bowl and common salt as a builder in the second.

4) Neutral nonionic detergent scouring, similar to "3" but using no soda ash.

5) Soap and soda scouring.

Full details of these experiments are given, and the results are shown in tabular form.

Solvent degreasing produced a higher yield of scoured wool than any of the aqueous methods. Differences of 0.5% to 1.4% of clean wool dry fiber were found when solvent degreasing was compared with traditional soap-soda scouring.

Neutral nonionic detergent scouring gave only 0.34% lower yield of dry wool than solvent scouring, but was the least efficient, as indicated by a high residue of grease content.

Simulated soap-soda and alkaline nonionic detergent scouring gave yields of dry wool even lower than the soap-soda method.

Evidence is presented by the author showing that the difference in yield of dry wool is due to dissolution of oxidized protein material from the tip portion of the wool fiber by hot alkaline scouring solutions.

The regain of scoured wool at standard conditions was found to vary with the method of cleaning. Wool cleaned by solvent or neutral detergent had a lower regain than that from alkaline scouring.

Thirty-four references to the literature are cited.

●

The Interaction between some Acid Wool Dyes and Nonyl Phenol-Ethylene Oxide Derivatives

Craven, B R and Datyner, A, *J Soc Dyers & Col 77, 304-12, July, 1961*

The use of surface-active agents (surfactants) in textile dyeing has become widespread in recent years, the most general claim made for their beneficial effects being that they assist in level dyeing. Various modes of action have been suggested for the achievement of level dyeing in different surfactant-dye-fiber systems, all relating to one or other of the properties peculiar to surfactants, which are discussed.

Combination of dye with ionic surfactants can take place when the two are oppositely charged, but the complex thus formed tends to flocculate. Complex formation can also occur between ionic dyes and nonionic surfactants, when flocculation does not take place.

The behavior of nonionic surfactants with respect to level-dyeing acid dyes on wool is not easily predictable. When mixtures of wool dyes are used, it is not unknown for some components to be affected strongly by the addition of a nonionic surfactant, whereas others are unaffected. 2:1 Metal-complex dyes, however, appear to exhibit some uniformity in behavior in presence of nonionic surfactants.

It has also been noticed that color changes occur on addition of certain nonionic surfactants to aqueous solutions of some dyes.

The aim of the present investigation was to determine whether the change in dyeing behavior of a dye, due to presence of a nonionic surfactant in the dyebath, could be correlated with changes in the absorption spectrum of the dye under conditions similar to those used in dyeing.

A number of acid dyes, all having essentially the same structure but with different end-groups, were selected for this work, and two nonyl phenol-ethylene oxide surfactants with approximately 9 and 29 ethylene oxide units respectively were added to the dyebaths. Dyeings were made on wool in neutral solutions in absence of salt, and were sampled at intervals between 55° and 100C°.

Solutions of the dyes were made, containing the same amounts of the surfactants, and their absorption spectra were measured in a Beckman spectrophotometer.

The results of both dyeing and spectrophotometric experiments showed that for all the systems examined there is an interaction between dye and nonionic surfactant, but there is a wide variation in the extent to which the various dyes react with a given surfactant.

In general, the uptake of dyes by wool from a neutral dyebath increases with the ratio of hydrophobic to hydrophilic parts present in the dye molecule (as deduced from the dye structures), a trend which is shown by the dyes selected for this investigation.

Further, the results indicate that the extent to which a surfactant modifies the dyeing behavior of a dye also increases as the hydrophobic nature of the dye increases.

The authors conclude that the complex formed between dye and surfactant acts as a "reservoir" from which dye is gradually released on heating the bath, by decomposition of the complex.

Thirty-one references to the literature are cited.

●

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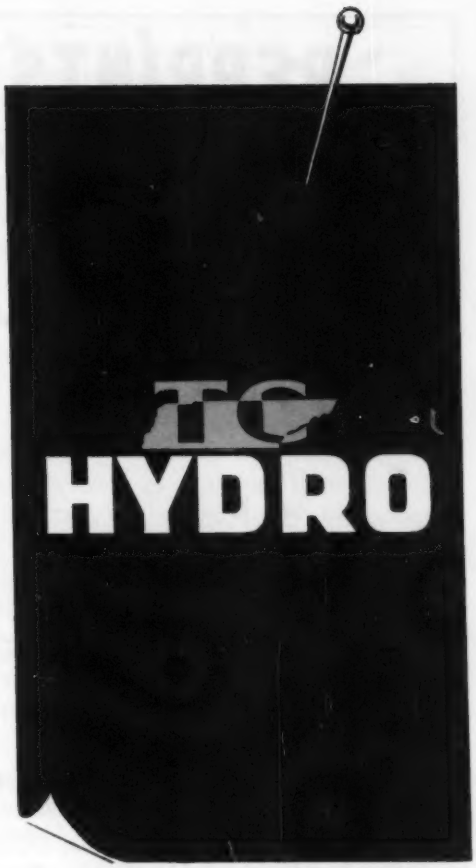

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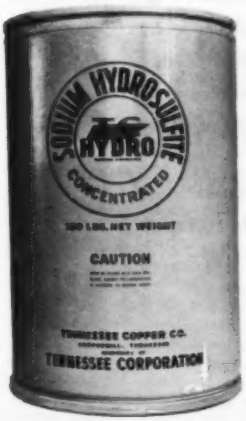

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Write: REPRINTS, American Dyestuff Reporter
44 East 23rd Street, New York 10, N Y

SEE FRONT COVER

Cibacron® Brilliant Red BD

...a NEW reactive dye for direct printing on natural or regenerated cellulosic fibers... offering excellent solubility without crystallization

CIBA extends dye performance beyond the little red school house concept. The famous Cibacrons – now numbering over two dozen – are the result of CIBA's traditional emphasis on research. Deeply rooted is the reputation of the Cibacrons for their original brilliance and fastness. They're doubly famous for their mature performance . . . they're versatile . . . they retain their brilliance . . . *they follow through!* Typical of other members of the growing Cibacron family, the new Cibacron Brilliant Red BD overcomes the enemies of follow-through brilliance. This new CIBA reactive dye offers excellent fastness to water and washing, perspiration and dry cleaning. Even normal dirt and grime – when removed by conventional methods – do not blemish the dye's original little-red-school-house complexion. Cibacron Brilliant Red BD offers unusually good solubility. Produces print pastes of very good stability without crystallization. Permits excellent fixation by normal printing applications. White discharge is obtainable in pastel shades. Cibacron Red BD is suitable for dyeing of fabrics which are to be resin finished, rubberized or P.V.C. coated. For complete details contact your nearest CIBA representative or CIBA Company Inc., Fair Lawn, New Jersey.

C I B A

ADR News Report:

General News of the Industry
Personalities in the News
New Products, Developments, Processes

Hylite

Proctor Chemical Co, Inc, Salisbury, NC, has announced the release of a new thermosetting resin, Hylite, which is said to enable the finisher to produce economically, excellent crease resistance or "wash-and-wear" effects without harming the lightfastness of most classes of dyestuffs. According to Proctor Chemical, of particular interest is the excellent protection Hylite gives to the original lightfastness ratings of the reactive dyestuffs.

•

Polasan Liquid Softeners

Onyx Chemical Corp, 190 Warren St, Jersey City 2, NJ is now offering a range of Polasan liquid, cold-water-soluble softeners and lubricants for the textile industry. The series is comprised of Polasan A, Polasan N and Polasan C, denoting the anionic, nonionic and cationic types respectively. The Polasans are said to be characterized by their ease-of-handling physical state combined with low cost, high degree of softening, antistatic effect and compatibility with a wide range of finishing materials, such as resins and builders.

Polasan and Polasan N reportedly have extremely high resistance to discoloration due to high-temperature processing.

The Polasans are said to offer the finisher a complete series of chemical types to fit the wide variety of finishing formulations encountered in today's mills, and to speed up bath preparation and minimize waste. The Polasans are immediately available for plant application.

Data file sheets on the series are available from Onyx on request.

•

Armosuls 16 and 18

Development of two new anionic surface-active agents, alpha-sulfopalmitic and alpha-sulfostearic acids, has been announced by Armour Indusrial Chemical Company.

Both acids, known as Armosul 16 and Armosul 18, respectively, are said to be difunctional compounds having a strongly acidic, sulfonic acid group and a weakly acidic, carboxylic acid group within the molecule. Both acids and various derivatives reportedly have unusual solubility and surface-active properties.

Known applications of Armosul acids are said to include improved detergent formulations at lower costs by combining alkali metal, ammonium, or substituted ammonium salts of sulfated aliphatic alcohols with similar salts of alpha-sulfonated fatty acids. In addition, it is claimed that improved detergency can be obtained by replacing some of the alkyl aryl sulfonates, which are relatively ineffective detergents, with a mixure of salts of Armosul acids and sulfated alcohols.

Armosul 16 and Armosul 18 are available in pilot-plant quantities. They are said to be off-white, slightly hygroscopic powders which are soluble in water and polar organic solvents.

•

Cibanone Grey P2R Double Paste

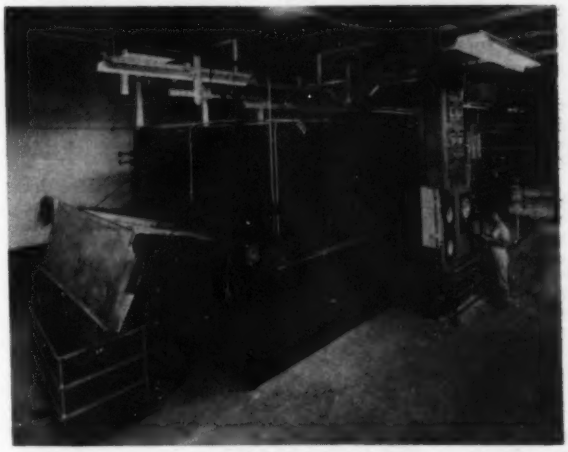
A clear, neutral-toned vat grey said to possess excellent fastness, Cibanone Grey P2R Double Paste is a new dye developed by Ciba.

It reportedly has been designed to dye grey shades uniformly with little or no shading colors added, for many current grey shades. Other grey shades may easily be produced by a wide range of shading components, it is claimed. Level dyeing of Cibanone Grey P2R Double Paste reportedly facilitate reproduceability.

Developed primarily as a yarn-dyeing color, Cibanone Grey P2R Double Paste is said to be suitable for application by conventional processes to cotton and rayon piece goods.

Of excellent build-up properties, Cibanone Grey P2R Double Paste has been realistically priced, it is stated.

Samples, technical bulletins and price information are available from Ciba Co, Inc, Fair Lawn, NJ.



This Saco-Lowell/Fleissner Dryer was recently installed at the Washington Mills Co, Mayodan, NC. The unit will be used primarily to dry tubular cotton knit goods, officials said.

Spanyl Colors

Dyes for spandex elastic fabrics are now being marketed by the Althouse Chemical Co, a division of the Crompton & Knowles Corp.

Called Spanyl, these new colors reportedly do not require any unusual dyeing procedures or techniques, but are applied according to standard practical dyeing procedures. The Spanyl series of dyes include: Spanyl Yellow 2GS; Spanyl Orange 2RS; Spanyl Maroon TRS; Spanyl Red 2BNS; Spanyl Blue 3RDS and Spanyl Grey NS.

It is claimed that the Spanyl dyes enable the dyer to obtain good union dyeing between the nylon and spandex, together with good fastness properties.

•

Marmix 16123

Development of a new latex, designed to improve both physical properties and performance in a wide range of products compounded from natural and synthetic rubber latices, has been announced by the latex and coatings department of Marbon Chemical Division of Borg-Warner Corp, Washington, W Va.

Introduced under the trade name of Marmix 16123, the new product is a high styrene terpolymer reinforcing latex which reportedly can be blended with other rubber and resin latices to provide improved base compounds. Said to be compatible with natural rubber, styrene-butadiene, nitrile and neoprene latices, Marmix 16123 Latex reportedly thickens easily, improves color stability, lowers process temperature requirement, and offers both stiffening and improved tensile advantages. The new modifier has a relatively low flux or fusion temperature, providing a margin of safety for plant processing, is is stated.

The Company further states that, by varying the level of the new latex, it is possible to modify a base compound to give finished products stiffness control, good age resistance, increased hardness and tensile strength, greater dimensional stability, tear resistance and higher compression modulus.

At modifications of 15% or greater, Marmix 16123 Latex reportedly imparts a degree of thermoplasticity to cured rubber compounds. This characteristic, according to Marbon, has particular value for products which must be embossed or post-formed. The new latex also is said to offer dependable compounding behavior and important reinforcing advantages when used in textile coatings, dipped or foamed latex goods, rug backings, paper products, and specialty coatings.

HFS Starch

A new, reportedly high-film-strength starch for warp sizing, called HFS Starch, is being introduced by National Starch and Chemical Corporation to the textile field.

HFS Starch reportedly has been chemically modified in a new way to produce tougher starch films with greater strength. Instron tests are said to have shown that the films are up to 30% stronger. The high film strength of HFS Starch has made efficient weaving practical with a lower size pick-up, it is claimed. It is also claimed that higher efficiency is also attainable with yarns made from low-grade fibers due to the superior binding qualities of HFS.

According to NSCC, a most desirable feature that is distinct with HFS Starch is its ability to resist viscosity breakdown on continued cooking. HFS Starch also is said to reduce viscosity breakdown due to excessive size circulation, pumping and other mechanical factors.

Films of HFS Starch reportedly absorb 30% less water than pearl starch, and it is suggested that this is an important factor to be considered by mills having humidity spray units, where droplets of water occasionally strike the warp or where there are variations in the percent relative humidity.

National Starch has prepared a new booklet describing this new unique warp sizing concept. Copies are available by writing the company at 750 Third Ave, New York, NY, Department T.

•

Twitchell 7428 Oil

A new economy-priced antistatic lubricant is offered by the Organic Chemicals Division of Emery Industries, Inc. The lubricant, Twitchell 7428 Oil, reportedly is designed for the production of popular-price yarns and fabrics, including blends.

Twitchell 7428 is said to offer effective static control, easy removal, and balanced penetrating and spreading properties. It reportedly may be used in all conventional application systems, or sprayed in emulsions as concentrated as a water-to-oil ratio of 2 to 1.

Full information on Twitchell 7428, as well as the other members of the Twitchell lubricant line, is contained in Technical Bulletin No. 414A. Copies may be obtained by writing Emery Industries, Inc, Dept 5, Carew Tower, Cincinnati 2, O.

•

Nerosol Black RF

The United States Patent Office in July issued a patent to the Organic Chemical Corporation for a new dyeing process capable of dyeing wool stock, tops, and piece goods.

The process is said to cut the wool-dyeing time from 3½ hours down to 1¼ hours. Nerosol Black RF reportedly enables scheduling three dyeing cycles instead of two.

This method, it is stated, allows more efficient carding and spinning, less card waste due to the better condition of the stock, and consequently, more efficient yarn production, plus added tensile strength.

Fastness properties reportedly remain unchanged. Nerosol Black RF is said to be adaptable for nylon, as well as wool and worsted.

For further information contact Organic Chemical Corp, 84 Valley St, East Providence, RI.

•

Palogen DP; Antifoam DP

De Paul Chemical Co, 44-27 Purvis St, Long Island City, NY, has announced the development of a new product, Palogen DP, to ensure level dyeing of texturized nylon filament yarns, such as Ban-Lon, Helanca, Tycora, etc, with acid dyestuffs.

Palogen DP reportedly has been widely tested and used successfully in many mills to overcome the barré effect in dyeing caused by knitting irregularities or uneven affinity of the various dyestuffs. Palogen DP reportedly enhances the migration properties of acid dyestuffs and slows down the exhaustion rate, thereby ensuring level dyeings. It is said to have excellent solubility and stability.

Antifoam DP is described by De Paul as "a new, highly effective, low-cost antifoaming agent, recently developed for all phases of textile processing operations, such as pad dyeing, jig dyeing, boil-off, scouring and finishing, etc." It will also, very effectively, control foam in the printing processes without affecting the viscosity of the printing paste, the Company states.

NAMES IN THE NEWS

Suter

Paciello

Meadows

Charles A Suter has been elected president of Geigy Chemical Corp, succeeding *William F Zipse*, president of the firm since 1943. Marking his 58th year of service with Geigy, Mr Zipse now becomes chairman of the executive committee.

Mr Suter had completed ten years with J R Geigy, SA in Switzerland prior to joining the United States company. Rising to vice president and director in 1943, he has been executive vice president since 1950.

•

Berkshire Color & Chemical Co, Reading, Pa, has named *Joseph A Paciello* manager of its Paper and Leather Division. Mr Paciello, who has had 22 years of experience in the pulp, paper and leather industries, was associated with E I duPont de Nemours & Co, Inc, for twenty years and then joined the Amalgamated Leather Co as assistant to the president.

•

Ernest L Frankl Associates Inc, 515 Madison Ave, New York 22, NY, has recently announced the appointment of *Robert H Meadows* of Charlotte, NC, as sales representative.

Mr Meadows will cover primarily the Southern States for the various European machinery accounts of Frankl, and he will make his headquarters in Charlotte.

Mr Meadows was previously associated with the Cosa Corp of New York.

•

The appointment of *Howard M Sprock Jr* as sales representative for Hycar special purpose rubber has been announced by B F Goodrich Chemical Co.

In his new position, Mr Sprock will be responsible for Hycar latex and dry rubber sales in southern Virginia and most of North Carolina. He has been active in this area for the past year promoting the sale of BFG Chemical products for the textile industry.

•

A J Farnworth, inventor of the Si-Ro-Set process for imparting permanent creases to pure wool trousers and durable pleats to women's wool skirts, has been appointed manager of the Technical Development Section of the Australian Wool Bureau.

Dr Farnworth was a principal research officer with the Commonwealth Scientific and Industrial Organization, Division of Textile Industry, at Geelong, Victoria prior to his new appointment, which took effect September 18.

As technical manager of the Australian Wool Bureau, Dr Farnworth will lead a team of technical officers whose responsibilities include the personal introduction of new wool processes to the textile industry.

•

U S Senator *John O Pastore*, of Rhode Island, has been named by the Textile Section of the New York Board of Trade to receive its Annual Award for 1961 in recognition of his keen understanding and loyal support of the Textile Industry.

Garret J Garretson II, vice president, Turner Halsey Co, and chairman of the Textile Section, has announced the award will be made at a luncheon on Thursday, November 9, at the Hotel Pierre.

•

George H Hotte has been appointed executive vice president of Texplant Corporation.

In this new position Mr Hotte's principal activity will be in Texplant's fibers, yarns and fabrics division.

Prior to joining Texplant Corporation Mr Hotte was director of fiber marketing for Allied Chemical Corp.

His headquarters will be Stamford, Conn.

Brigadier General Merrill L Tribe, commanding general of the U S Army Quartermaster Research and Engineering Command, Natick, Mass, presents a Department of the Army Research and Development Achievement Award to Frank J Rizzo, chief of the Command's Textile Dyeing Laboratory, at a special retreat ceremony at the Natick Headquarters.

The Department of the Army recently announced that *Frank J Rizzo*, chief of the textile dyeing laboratory, Quartermaster Research and Engineering Command, Natick, Mass, is a winner of the first annual Army Research and Development Achievement Award.

Mr Rizzo was honored for guiding the development of a fully automated instrument which is said to measure and record color differences numerically with a degree of accuracy never before attained. The color meter assures exact color matching and also records data that can be used for future color-matching requirements.

Brigadier General Merrill L Tribe, commanding general, QM R&E Command, presented the Army's newest award to Mr Rizzo at a special retreat ceremony held at the military scientific installation at Natick.

The Army R&D Award consists of a bronze wall plaque and a lapel pin. It is based on accomplishments which establish a scientific basis for subsequent technical improvement of military importance, materially improves the Army's technical capability, or contributes materially to national welfare.

Earlier this year for his work on the color meter, Mr Rizzo received the QM R&E Command Research Directors' Award for Development.

●

Godfrey S Rockefeller, president, National Association of Finishers of Textile Fabrics, New York, will be general chairman of the tenth annual Chemical Finishing Conference.

The Conference, sponsored by the National Cotton Council, will be held at the Sheraton Park Hotel in Washington, DC, November 8 and 9.

Mr Rockefeller, who is also president of Cranston Print Works Co, New York, will serve as conference chairman for the second successive year.

Session chairmen for the conference will be: *Stephen J Kennedy*, research director, Textile Clothing and Footwear Section, Army Quartermaster Corps, Natick, Mass; *Giuliana Tesoro*, assistant director of research, Central Research Laboratory, J P Stevens & Co, Garfield, NJ; and *Henry Tovey*, National Cotton Council, Washington DC.

●

Raytex Chemical Corp, Allentown, Pa, has announced the addition to its staff of *F L Young* as chief chemist and technical consultant in development and textile research. Mr Young joins Raytex with many years of experience, having worked with General Dyestuff Co and for a number of years having directed dye applications. He spent a number of years in development research of Vicara bleaching, dyeing and processing. In 1941, Mr Young is said to have developed a process for dyeing acetate in fast colors in a water medium which at that time replaced 95% of the very costly alcohol procedures then in use.

●

Appointment of *Paul B Cornell* as assistant manager of Allied Chemical's Solvay Process Division plant at Moundsville, W Va, has been announced.

Mr Cornell moves up from the post of assistant manager of Solvay's Brunswick, Ga, plant.

Szczepanik

Rogers

Hamilton Chemical Corp, Lowell, Mass, has announced the appointment of *Henry M Szczepanik* as vice president and director of research.

Mr Szczepanik joins Hamilton Chemical Co after having served as a research chemist at Polyvinyl Chemicals, Inc, Peabody, Mass, developing styrene and acrylic emulsion polymers.

He will direct the research and development activities of Hamilton Chemical's Polymer Division, including its new line of crosslinking acrylics for the textile and paper fields.

●

Thomas F Rogers, formerly with Cargill, Inc, of Norfolk, Va, recently joined the Virginia Chemicals & Smelting Company's staff as a technical service representative.

●

OBITUARY

HAROLD W COWARD, president of Alliance Color & Chemical Co, passed away on August 11th at Doctor's Hospital in New York. He was active in the chemical processing industry for over thirty years. After his graduation from the Univ of Pennsylvania, he was employed by the National Aniline Div of Allied Chemical Corp in Buffalo, NY, and later by Interchemical Corp in New York. He was one of the founders of Alliance in 1946.

●

PAUL A DUNKEL, board chairman of the Paul H Dunkel Co, chemical importers of New York and Jersey City, died recently in St Luke's Hospital. He was 67.

●

ROBERT M AUDE, president of the Heyden Chemical Div, Heyden Newport Chemical Corp, died Sunday, August 20, at his home at 11 Lillane Lane, Upper Saddle River, NJ, after a short illness. He was 47 years old.

Mr Aude was born in Green Bay, Wis, and was a graduate of the University of Wisconsin. He was active in the chemical industry for more than 20 years.

Prior to joining Heyden, he was with Monsanto Chemical Co. In the eight years that he was with Heyden, he was successively manager of the Corporation's Garfield and Fords, NJ, plants; director of sales planning and coordination in New York; and vice president and general manager of Heyden Chemical Division, prior to his appointment as president of this Division in May 1960.

●

ARTHUR D CHAMBERS, 91, for whom the Du Pont Company's Chambers Works at Deepwater Point, NJ, was named, died in the Wilmington, Del, Memorial Hospital on August 28 after a short illness.

Dr Chambers, who lived in Wilmington, retired from the Du Pont Company in 1944 after 47 years' service. The Chambers Works, for which his recommendations were largely responsible, was given that name to honor him at the time of his retirement.

A native of Ontario, Canada, he was a graduate of Woodstock College and Columbia Univ. He received his PhD in chemistry from Johns Hopkins Univ.

After graduating from Johns Hopkins, he was employed by the Standard Oil Co, but joined the Du Pont Co as assistant superintendent of the Ashburn, Mo, dynamite plant in 1897.

In 1915 he was transferred to the Development Department in Wilmington and joined the Miscellaneous Manufacturing Department, of which Lammont du Pont was manager, in 1917.

In his position, Dr Chambers was one who was instrumental in the development of synthetic organic chemicals which freed this country from dependence on Europe for dyestuffs.

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ATTRACTIVE OPPORTUNITIES exist for sales representatives experienced in handling dye and/or textile auxiliary lines. Positions available in both Charlotte and Atlanta territories. Send a brief resume outlining education and vocational background to: Sales Manager, ARNOLD, HOFFMAN & CO., INC., 55 Canal Street, Providence, R. I. A subsidiary of—Imperial Chemical Industries Limited. Your reply will be handled in confidence.

WANTED: YARN DYER for commission dyehouse in New England area. Experience on all fibers and on yarn in all forms required. Give complete information and salary requirements. Write Box No. 225

WANTED: TEXTILE CHEMIST, for synthetic piece dye plant. Minimum 2 years experience required. Salary commensurate with experience. Submit resume and salary requirements with first letter. Replies confidential. Write Box No. 226

WANTED: CONSULTANT — CHEMICAL ENGINEER OR TEXTILE ENGINEER, production experience in dyeing, finishing and printing for staff of largest international textile consulting organization. Excellent growth opportunity. Send details of background, references, and salary status. Write Box No. 227

WANTED: TEXTILE CHEMIST for dye application to natural and synthetic fibers. Experience in dye laboratory and/or mill helpful, but not required. Position offers opportunity in application-research and standardization work with dyestuff manufacturer. May also require technical service work. Location Metropolitan area. Send resume, references and salary requirements to Box No. 228.

SALESMEN WANTED: Several new territories currently open with expanding, old-established organization. Manufacturers of softeners, resins, sulfonates and a diversified list of highly specialized chemical auxiliaries. Man must have a practical background and good sales personality. Excellent opportunity for good income with a stable, progressive, medium sized company. Submit full background and references. Our employees know of this ad. Write Box No. 229

WANTED: YOUNG MAN with knowledge of dyeing and finishing who would prefer selling to present position. Training program prior to assuming position in South. Salary, traveling expenses, and commission. Write Box No. 230

• PERSONNEL AVAILABLE •

POSITION WANTED: Perfectionist—14 years experience as Chemist, Plant Superintendent, Dyer and Technical Sales. Desire challenging position in plant currently not satisfied with present efficiency, costs and quality. Write Box No. 217

POSITION WANTED: GRADUATE CHEMICAL ENGINEER AND TEXTILE CHEMIST. 20 years diversified experience academic work in chemistry research and development in dyeing and finishing production and supervision in Resin and Starch finishing. Desires position as Finisher or Executive. Write Box 220

POSITION WANTED: TEXTILE CHEMIST—TECHNOLOGIST. Ph.D., fifteen years experience in research, development, mill practice, administration. Special experience with pile fabrics, latices, elastomers, non-wovens, synthetic fiber applications. Write Box No. 224

1962

AMERICAN DYESTUFF REPORTER

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JULY	9-23
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SPECIFICATIONS . . .

Information should include the trade name of the product and a BRIEF summary of its uses and properties. Please type (double-spaced) the information for each product on a separate sheet of paper (8½ x 11). If illustrations are necessary, glossy photos or black ink drawings should be furnished. It the trade name is registered or the product is patented and you wish these facts indicated, please so note.

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In order to insure that your new products for wet-processing may be listed in this PROCESSING REVIEW NUMBER of December 11, information concerning them should be in our office NO LATER THAN NOVEMBER 1st. *It is important that you list only those products which have been brought out since November,* 1960. Information should be addressed to The Editor, American Dyestuff Reporter, 44 East 23rd Street, New York 10, N.Y.

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Metalsmiths Div., Orange Roller Bearing Co., Inc.
Mettler Instrument Corporation
Morningstar-Paisley, Inc.
Morton Salt Co.

National Aniline Division
Allied Chemical Corp. 20
National Starch and Chemical Corporation
Nopco Chemical Company
Nyanza Color and Chemical Co.

Olin Mathieson 29
Onyx Chemical Company
Organic Chemical Corporation

Philadelphia Quartz Co.
Photovolt Corp.
Plastics Division, Allied Chemical Corp.
Putnam Chemical Corp. 19

Reading Scientific Company
Reiner, Incorporated, Robert
Rohm & Haas Co.
Royce Chemical Company
Rumford Chemical Works

Sandoz, Inc.
Scholler Bros., Inc. 17
Seydel-Woolley & Co. 16
Sou-Tex Chemical Company, Inc.
Southern Dyestuff Company
Spencer Chemical Company
Sun Chemical Company

Tanatex Chemical Corp. Second Cover
Taylor Instrument Companies
Tennessee Corp. 53
Tex-Chem Co.
Toshin Kogyo Co., Ltd. 32
Turbo Machine Company

Union Carbide Corp.

Valchem 59
Verona Dyestuffs
Virginia Chemicals & Smelting Company 12

Wallace & Tiernan, Inc., Harchem Division
Wallerstein Company
West Indies Chemical Works, Ltd., The 62
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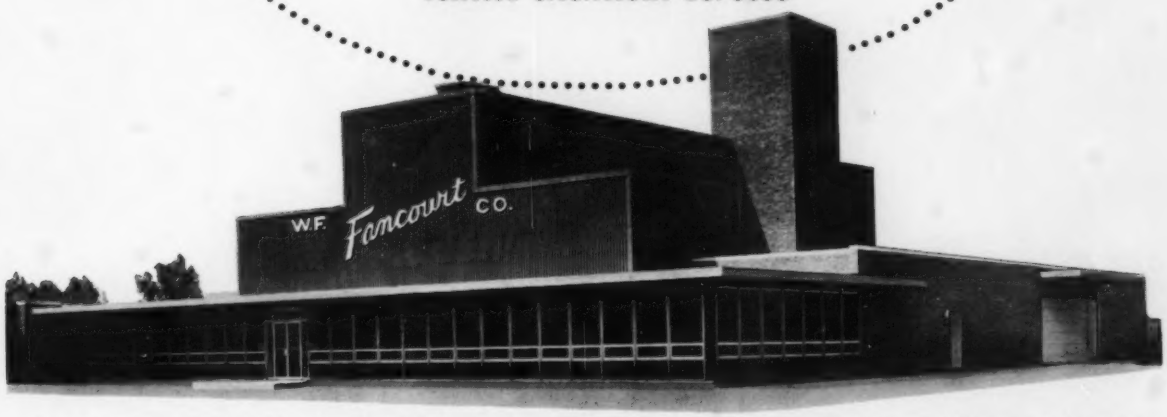

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Canadian Office—Chemtex Products, Ltd.
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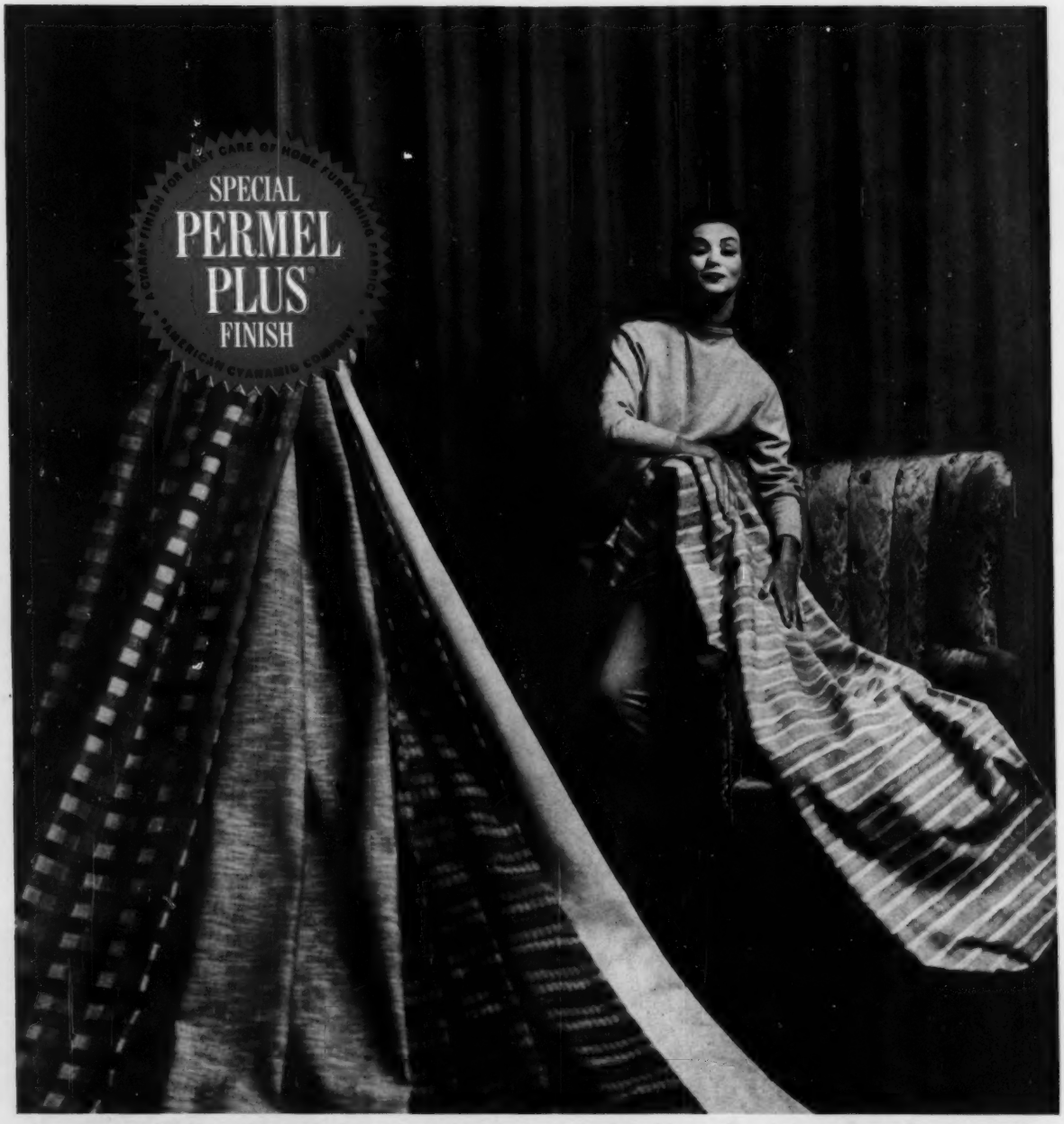

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